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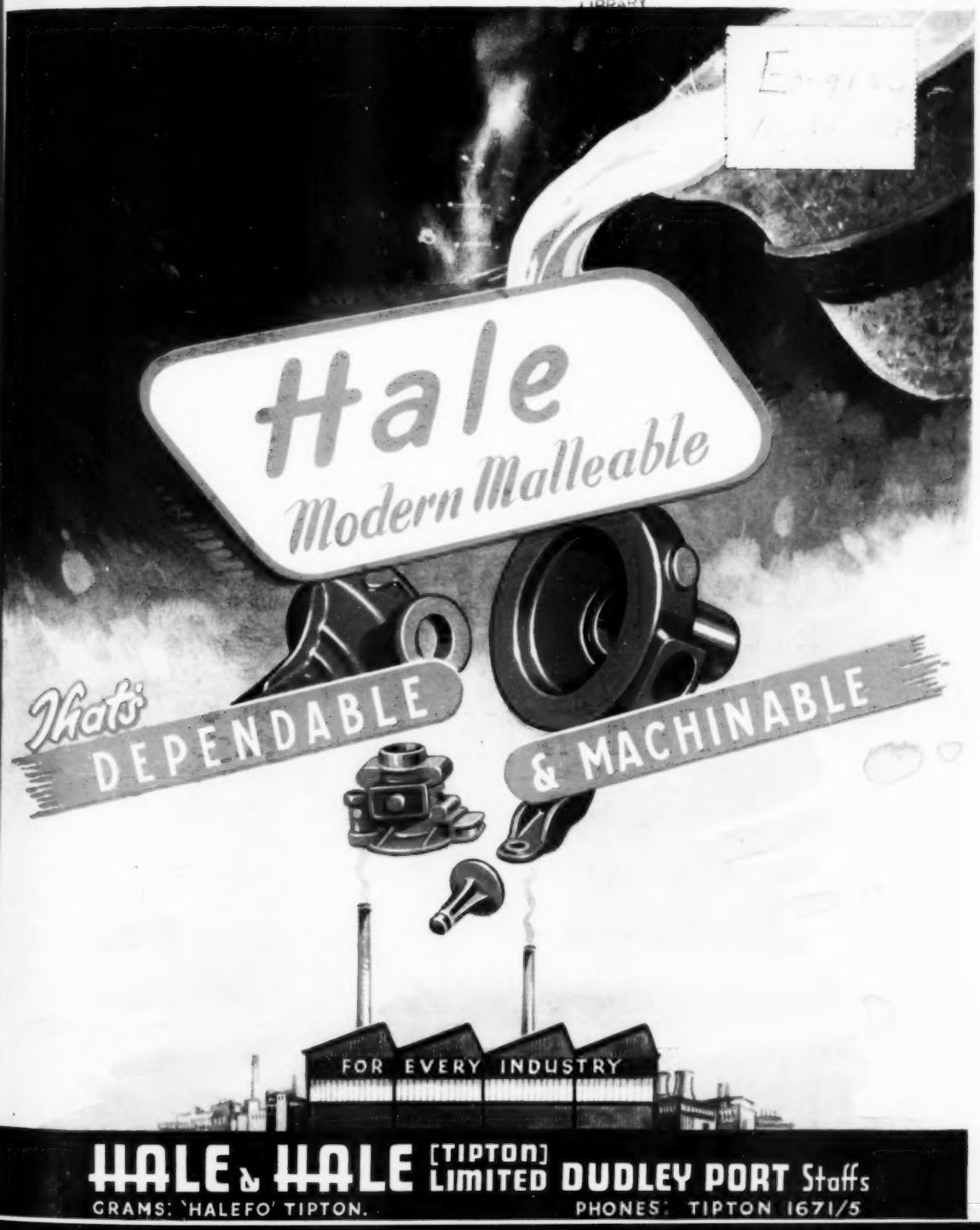

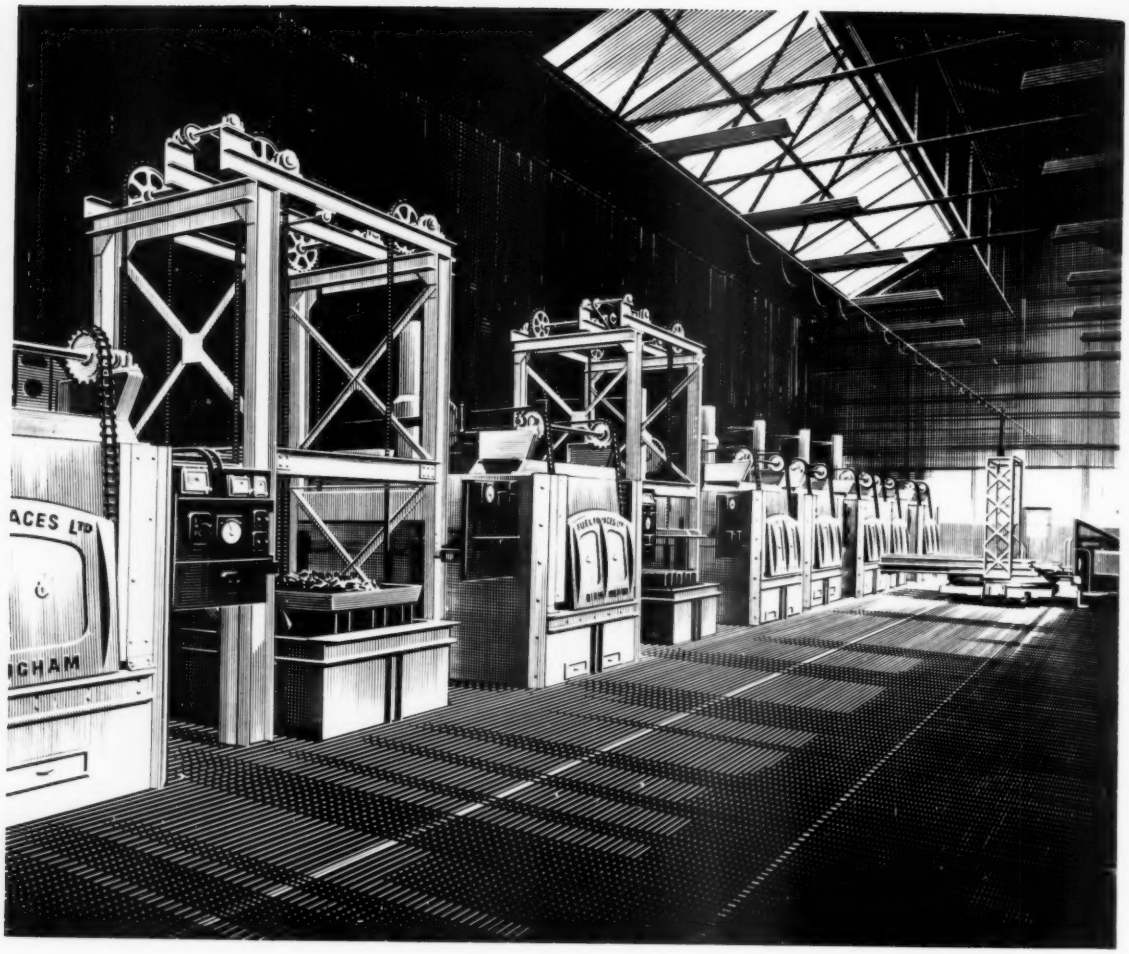

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THE BRITISH JOURNAL OF METALS
INCORPORATING THE METALLURGICAL ENGINEER

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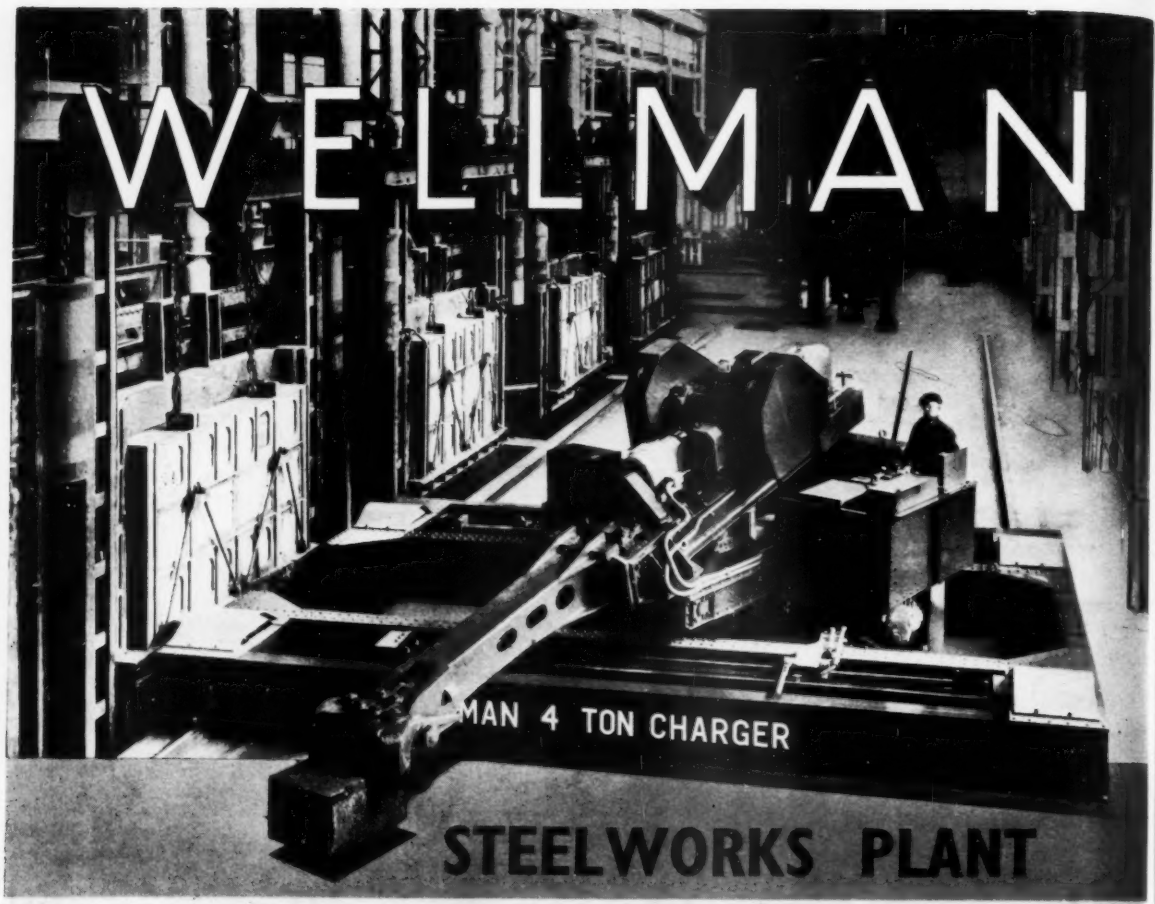

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METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER,"

OCTOBER, 1958 Vol. LVIII. No. 348

Free Trade in Aluminium

ONE of the minor headaches associated with the publishing of an article on aluminium and its alloys, at the end of which are listed references to papers published on the two sides of the Atlantic, arises from the difference in spelling of the word "aluminium" in Britain and North America. As many readers are no doubt aware, the American spelling is "aluminum"—incidentally, the name by which the metal was first known—and there is a natural tendency to correct what appear to be spelling mistakes in the titles of the American or Canadian papers. In time one becomes accustomed to the omission of the "i" and accepts the American spelling. Having done so, it is rather confusing to find that there exists a Canadian firm, Aluminium Limited, which uses the English spelling in its name. Ownership of the company is largely American, and its main production facilities are located in Canada, being operated by the fully owned subsidiary, Aluminum Company of Canada, Limited. How is it, then, that the American spelling is not used? The answer to this question was given recently by Mr. Nathaniel V. Davis, President of Aluminium Limited, when speaking to The Executives' Club of Chicago: "Because we do business in many parts of the world, Aluminium Limited adopted in its name the spelling of the word for the metal used virtually all over the world outside North America."

About 20% of the primary aluminium produced by all the nations outside the Communist bloc moves into international trade, the remaining 80% staying within its country of origin. Because of the possession by them of natural facilities favourable to the production of aluminium, namely abundant hydro-electric power resources close to deep-water ports, the traditional exporting nations to the markets of the free world are Canada and, to a lesser extent, Norway. Some 85% of Canadian aluminium production is for export, and it is not surprising therefore, that Mr. Davis chose to speak on the subject of free world trade in aluminium, for Mr. Davis's company is vitally concerned with the status of aluminium in world commerce.

Since 1945 there has been a rapid growth in the production and consumption of aluminium, which has led to considerable capital investment in plants, such as Kitimat and Baie Comeau, for producing the primary metal. This expansion has resulted in increased international trade in aluminium, and many of the tariffs and other forms of restriction which formerly obtained have been reduced or removed altogether. During the first decade of the post-war period this trade was strong and healthy, and there was a shortage of primary metal for a considerable period. During the last two years, the situation has changed: in the first place, production capacity caught up with demand; and in the second, the demand ceased to increase as the recession started. The net result is that there is a temporary surplus of production capacity, and today producers who are not traditional exporters are taking an active interest in international trade. The consequence of these conditions is that the price structure has weakened, but, as Mr. Davis pointed out, difficult as these developments are for the industry, they are merely the normal economic corrective forces designed to bring about most quickly a return to greater volume of consumption and a more satisfactory return on invested capital. Two facts are, however, causing concern in the industry, particularly in Canada: they are the revival of protectionist theories, and the Russian offers of primary metal on several world markets.

Primary aluminium is inherently an international commodity, and Mr. Davis emphasised that the various international components which go into its production make the industry unsuitable for narrow regional restrictive practices. For instance, Canada has no bauxite—the basic raw material—and its smelting plants receive the ore from the Caribbean, South America and Africa. Similarly, United States producers rely mainly on Caribbean and South American sources and are turning additionally to Africa and even the Far East.

Strong groups on both sides of the Atlantic are urging higher tariffs. In the United States, the major producers of primary metal jointly petitioned the Federal Government for increased protection against imports, and although no decision has yet been reached in respect of the six-nation area of the European Common Market, reports indicate that the three producing countries are promoting the erection of a high tariff to prevent imports of aluminium from outside areas. Aluminium is in competition with wood, plastics, steel and other metals, and is subject to the same economic laws as they are. The choice between aluminium and a competitive material is frequently governed by price, and if import restraints are built into the aluminium industry, the competitive position of aluminium can only be adversely affected by the resulting higher prices. "Aluminium," said Mr. Davis, "is one of those materials which, because of its virtually unlimited mineral resources and its wide application, can be used to build better lives for the people of the world. It would be regrettable if this challenge were forfeited by burdening the industry with artificial restrictions."

On the other hand, Mr. Davis felt there were circumstances in which protective tariffs could be used to advantage. During the past eighteen months, Russian primary aluminium has been offered on several international markets, and although the quantity of Russian metal sold has been small—about 1% of the free world supply—the terms of sales, which have included guaranteed price differentials, have had a somewhat disrupting effect on international markets. If this turns out to be a Russian weapon for use in the economic war, it will be necessary for the governments of the free world to take suitable measures to prevent damage to a vital private industry.

Meeting Diary

9th October

Incorporated Plant Engineers, Glasgow Branch. "Corrosion," by F. S. BRICKNELL. Scottish Building Centre, 425/427, Sauchiehall Street, Glasgow. 7-15 p.m.

Institute of Fuel, North Western Section (Merseyside Sub-Section). "Shell Boilers and their Maintenance," by J. N. WILLIAMS. Liverpool Engineering Society's Rooms, 9, The Temple, Dale Street, Liverpool. 7 p.m.

Liverpool Metallurgical Society. Presidential Address. "Alternative Methods of Iron and Steel Production," by DR. C. BODSWORTH. Library, Dept. of Metallurgy, The University of Liverpool, 146, Brownlow Hill, Liverpool, 3. 7 p.m.

13th October

Institute of Welding, Manchester and District Branch. "Welding as a Career for Adventure," by G. G. MUSTED. Small Hall, Technical College, Bolton. 7-15 p.m.

14th October

Sheffield Metallurgical Association. "New Developments in Steelmaking, with some Reference to Refractories Problems," by J. S. CURPHEY. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

15th October

Incorporated Plant Engineers, Kent Branch. "Ultrasonics in Industry," by C. B. ABEL-HARRY. Kings Head Hotel, High Street, Rochester. 7 p.m.

Society for Analytical Chemistry, Scottish Section. "The Determination of Acidity in Dark Lubricating Oils," by DR. W. GIBB and H. GIBSON; "Volumetric Analysis of Stannous and Total Tin in Acid-Soluble Tin Compounds," by J. D. DONALDSON and W. MOSER. Kenilworth Hotel, 5, Queen Street, Glasgow, C.1. 7.15 p.m.

16th October

Institution of Mining and Metallurgy. "Sinking No. 2 Shaft, Harmony Gold Mining Co., Ltd., Orange Free State," by S. C. NEWMAN; "The Micropanner—An Apparatus for the Gravity Concentration of Small Quantities of Materials," by L. D. MULLER. Geological Society of London's Rooms, Burlington House, Piccadilly, London, W.1. 5 p.m. Tea will be served from 4-30 p.m.

17th October

West of Scotland Iron and Steel Institute. Presidential Address, by DR. H. HARRIS. 39, Elmbank Crescent, Glasgow. 6-45 p.m.

20th October

Institute of Marine Engineers, West of England Section. "Metal Spraying and Synthetic Rubber Coating for the Prevention of Corrosion," by J. H. PAYNE. Grand Hotel, Bristol. 7.30 p.m. All visitors will be welcome.

North East Metallurgical Society. "High Strength Weldable Steels," by DR. K. J. IRVINE. Cleveland Scientific and Technical Institution, Middlesbrough. 7-15 p.m.

Sheffield Society of Engineers and Metallurgists. "Atmospheric Pollution in the Iron and Steel Industry," by J. H. FLUX and F. A. GRAY. The University Building, St. George's Square, Sheffield. 7.30 p.m.

21st October

Institution of Industrial Safety Officers, Manchester and District Branch. "Radioactive Hazards in Works Uses," by A. C. FARTHING. Board Room, Gaddum House, 16–18, Queen Street, Manchester. 6-30 p.m.

Sheffield Metallurgical Association. "The Determination of Gases in Metals by the Microvacuum Fusion Method," by E. BOOTH; "The Determination of Oxygen and Hydrogen in Steel," by C. E. A. SHANAHAN. **Joint Meeting with the Society for Analytical Chemistry.** B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m. The meeting will be preceded at 2.30 p.m. by a visit to the Laboratories.

22nd October

Manchester Metallurgical Society. "Martensitic Transformations," by DR. J. W. CHRISTIAN. Manchester Room, Central Library, Manchester. 6-30 p.m.

Society of Chemical Industry, Corrosion Group. "Cavitation Damage," by D. J. GODFREY. Society of Chemical Industry, 14, Belgrave Square, London, S.W.1. 6-30 p.m.

27th October

Incorporated Plant Engineers, West and East Yorkshire Branch. "Atomic Energy," by R. F. JACKSON. Fuel Department Lecture Theatre, New Building, Leeds University. 7-30 p.m.

28th October

Sheffield Metallurgical Association. "Some Aspects of Corrosion in the Canning Industry," by DR. H. LIEBMANN. **Joint Meeting with the Sheffield Section of the Royal Institute of Chemistry.** B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

4th November

Sheffield Metallurgical Association. "Furnace Design and Steelmaking Techniques," by C. HOLDEN. **Joint Meeting with the Iron and Steel Institute and the Sheffield Society of Engineers and Metallurgists.** B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

5th November

Institute of Welding, Manchester and District Branch. "Residual Stresses—Do They Matter?" by DR. L. E. BENSON. Reynolds Hall, College of Science and Technology, Manchester. 7-45 p.m.

Manchester Metallurgical Society. "Temperature Measurement," by J. A. HALL. Manchester Room, Central Library, Manchester. 6-30 p.m.

Society for Analytical Chemistry. "Volumetric Analysis of Stannous and Total Tin in Acid-Soluble Tin Compounds," by J. D. DONALDSON and W. MOSER; "Modifications to the Unicam SP 500 Spectrophotometer for Single-Beam Recording," by D. D. SHREWSBURY; "*o*-Dithiols in Analysis"—Part VIII; "The Use of the Zinc Salt of Toluene-3 : 4-dithiol in the Field Testing of Ores," by R. E. D. CLARK and C. E. TAMALE-SSALI. Meeting Room of the Chemical Society, Burlington House, London, W.1. 7 p.m.

6th November

East Midlands Metallurgical Society. "Titanium—A Broad Survey," by P. L. TEED. College of Art, Green Lane, Derby. 7.30 p.m.

Incorporated Plant Engineers, Southern Branch. A demonstration under workshop conditions with explanatory talk of electric welding. Chandler's Ford, Southampton. 7-30 p.m.

New Brayshaw Companies

As from the beginning of August, the Industrial Furnaces and Engineers' Tools Divisions of Brayshaw Furnaces and Tools, Ltd., Manchester, are trading as separate companies under the names of Brayshaw Furnaces, Ltd., and Brayshaw Tools, Ltd. This change, which takes place in the Diamond Jubilee year of the foundation of the original company, has been adopted in view of the continuous development and expansion of both divisions. The registered office of the two companies will remain at Belle Vue Works, Manchester, but a branch office for Brayshaw Furnaces, Ltd., has been opened at 84 Blonk Street, Sheffield, 3, and additional facilities for furnace repair and service are provided by a repair and service depot now operating at Northfield Road, Rotherham.

Araldite Price Reductions

SUBSTANTIALLY lower prices for Araldite epoxy resins have been announced by CIBA (A.R.L.), Ltd., (formerly Aero Research, Ltd.), the first organisation to manufacture epoxy resins in Great Britain. The reductions have followed immediately upon the completion of a new epoxy resin factory at Duxford with a greatly increased production capacity.

Some Observations on the Fatigue Behaviour of Aluminium-7% Magnesium in Various Conditions of Heat Treatment

By C. A. Stubbington, A.I.M.

Metallurgy Department, Royal Aircraft Establishment

Grain size is important in determining the fatigue properties of aluminium-7% magnesium, extruded material with fine grain size having better fatigue properties than solution treated material—in which grain growth had occurred—in all aged conditions. As-extruded aluminium-7% magnesium gave a steel type S-N curve, but any form of heat treatment, whether ageing or solution treatment, produced S-N curves typical of those obtained on other aluminium alloys. Cold working improved the fatigue properties of solution treated material. Partial intercrystalline fatigue failure may occur in materials failing in a transcrystalline manner under unidirectional stress.

IT has recently been suggested[1] that materials which show a sharp knee in their S-N curves are those which strain age during fatigue. Strain ageing is most marked in those materials which exhibit a sharp yield point, and mild steel is a good example of material of this type. A study[1] of the influence of strain ageing on the shape of the S-N curve in mild steel showed that removal by suitable heat treatment of carbon and nitrogen, elements normally associated with the yield point and strain ageing phenomena, tended to shift the knee of the S-N curve to longer life. It was suggested that the knee might be eliminated completely if these elements were sufficiently reduced. The strain ageing phenomenon has also been observed in aluminium-magnesium alloys, and in these alloys the well defined yield point can be removed by a heat treatment which increases the grain size. It was therefore decided to investigate the effect of grain size and ageing condition on the fatigue behaviour of aluminium-7% magnesium with particular reference to the relation between the shape of the S-N curve and that of the stress-strain curve.

Material Used

The alloy used in the investigation was made from superpurity aluminium and magnesium, and was chill cast in cast iron moulds into 1·5 in. diameter billets. These were homogenized at 420° C. for 16 hours quenched into cold water, and extruded in a small experimental

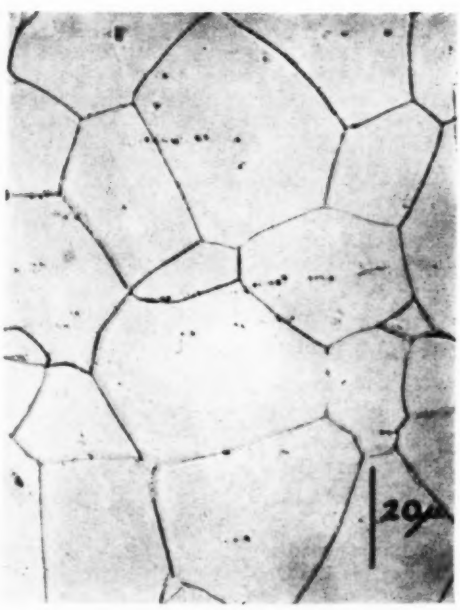

Fig. 1.—Microstructure of aluminium-7% magnesium, extruded and aged for 1 day at 150° C.

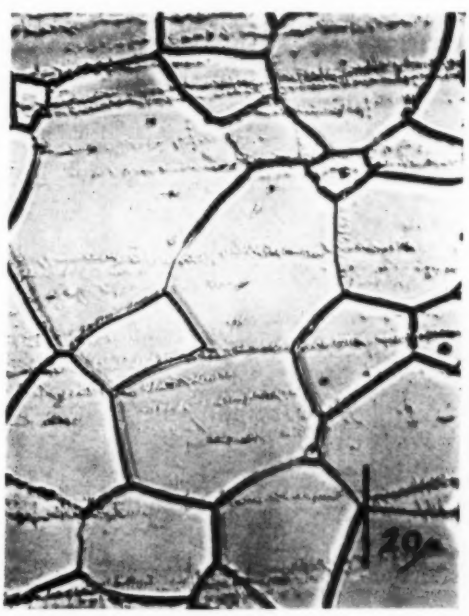

Fig. 2.—Microstructure of aluminium-7% magnesium, extruded and aged for 15 days at 150° C.

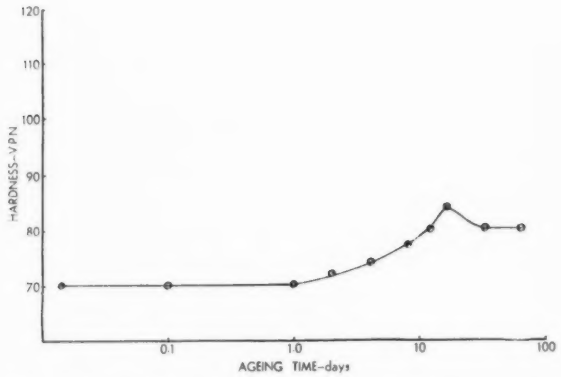

Fig. 3.—Ageing curve for aluminium—7% magnesium, solution treated at 420° C., cold water quenched and aged at 150° C.*

extrusion press. For the metallographic part of the investigation, the alloy was extruded in the form of rectangular strip of 0·15 in. thickness and 0·437 in. width. For the quantitative work, 0·3 in. diameter rod was extruded as a convenient size for the production of Rolls Royce fatigue specimens and 0·25 in. B.S.F. tensile test pieces.

Examination of Structures Produced by Heat Treatment and Cold Working.

It was found that the alloy had recrystallized during extrusion to produce a mean grain diameter of 0·023 mm. Solution treatment of this structure at 420° C. for 16 hours produced considerable grain growth and a mean grain diameter of 0·13 mm. The change in microstructure produced by ageing at 150°C. was then examined for both extruded and solution treated material. In the as-extruded condition there was no evidence of precipitation at the grain boundaries, but ageing for 2·4 hours at 150°C. produced some grain boundary precipitation. With increase in ageing time at 150°C., further precipitation occurred, and after 15 days at 150°C. the grain boundaries were completely outlined. Ageing for 15 days at 150°C. also produced some coarse precipitation within the grains on pre-existing stringers, which acted as nuclei. Fig. 1 shows the microstructure after ageing for 1 day at 150°C.,

* After Farmery and Evans, *J. Inst. Metals*, 1955–6, **84**, 413.

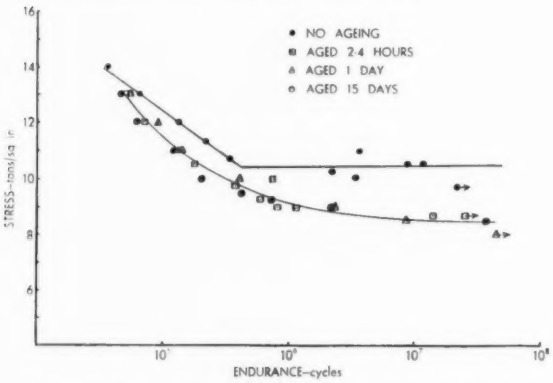

Fig. 5.—S-N curves for aluminium—7% magnesium, as-extruded and aged for varying periods at 150° C.

Fig. 4.—Microstructure of aluminium—7% magnesium, solution treated and cold rolled.

and Fig. 2 after ageing for 15 days at 150°C. Specimens which had been solution treated for 16 hours at 420° C., cold water quenched, and aged at 150° C., behaved in a similar manner. The ageing curve at 150° C. for solution treated and cold water quenched aluminium-7% magnesium is shown in Fig. 3. The fatigue and tensile properties for heavily cold worked aluminium-7% magnesium were also determined. To produce the necessary Rolls Royce fatigue specimens and 0·25 in. B.S.F. tensile test pieces, 0·625 in. diameter extruded bar was solution treated at 420°C. for 16 hours, cold water quenched, and cold rolled to a 0·3 in. square section. The longitudinal grain structure of material in this condition is shown in Fig. 4, from which it can be seen that considerable grain fragmentation has occurred.

Fatigue Tests

Rolls Royce rotating cantilever machines were used for these tests. S-N curves were determined for material in the following conditions :—

(i) Extruded condition.
(ii) Extruded, aged 2·4 hours at 150°C.
(iii) Extruded, aged 1 day at 150°C.
(iv) Extruded, aged 15 days at 150°C.
(v) Solution treated 420°C., cold water quenched.
(vi) Solution treated 420°C., cold water quenched, aged 2·4 hours at 150°C.
(vii) Solution treated 420°C., cold water quenched, aged 1 day at 150°C.
(viii) Solution treated 420°C., cold water quenched, aged 15 days at 150°C.
(ix) Solution treated 420°C., cold water quenched, cold rolled 75%.

When solution treatment was necessary, this was

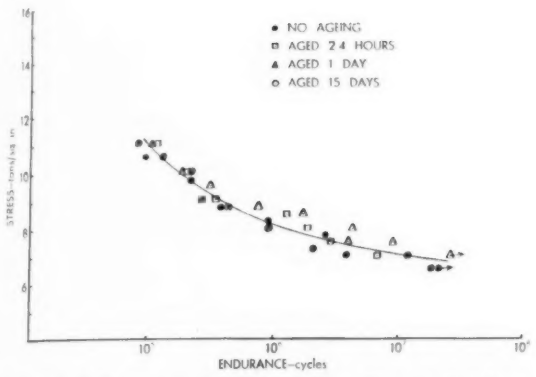

Fig. 6.—S-N curve for aluminium-7% magnesium, solution treated at 420° C. for 16 hours, quenched in cold water, and aged for varying periods at 150° C.

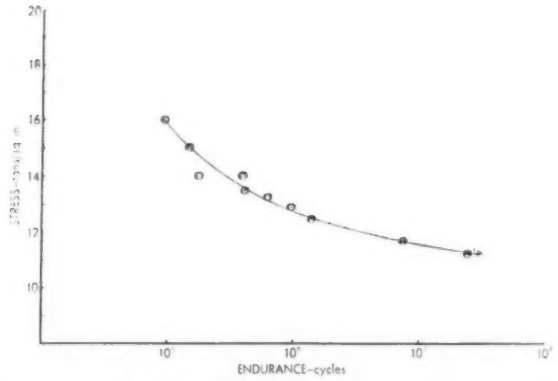

Fig. 7.—S-N curve for aluminium-7% magnesium, solution treated and cold worked 75% reduction.

carried out in argon for 16 hours. Specimens were then quenched into cold water. The S-N curves for extruded material in various conditions of heat treatment are shown in Fig. 5. It can be seen that in the as-extruded condition the shape of the curve is similar to that obtained for normalised mild steel, although the knee occurs at 5×10^5 cycles as opposed to 5×10^6 cycles for mild steel. Another similarity between the two materials is that the ratio of fatigue limit at 10^7 cycles to ultimate tensile strength (fatigue ratio) is of the order of 0·5. Ageing for 2·4 hours at 150° C., however, altered the shape of the curve by removing the well defined knee and lowered the fatigue limit at 10^7 cycles by 1·75 tons sq. in. and the fatigue ratio to 0·42. It can be seen from Fig. 5 that there is no significant difference in the fatigue behaviour between any of the aged conditions, despite the very considerable change in microstructure produced by prolonged ageing.

The S-N curve obtained on material fatigued in the solution treated condition and after solution treatment followed by various periods of ageing at 150°C. is shown in Fig. 6. It can be seen that the fatigue limit at 10^7 cycles is further reduced to 7·0 tons sq. in., and that there is little difference between the various conditions. The best fatigue properties for solution treated material, however, appear to occur after ageing for 1 day at 150°C. Ageing solution treated material for 15 days at 150° C. produced a fatigue ratio of 0·32, compared with 0·42 for the extruded and aged material.

The S-N curve obtained on material fatigued in the cold worked condition is shown in Fig. 7.

Fig. 8.—Photomicrograph showing transcrystalline failure with localised deformation in aluminium-7% magnesium specimen fatigued in the as-extruded condition.

Fig. 9.—Photomicrograph showing some intercrystalline failure in the predominantly transcrystalline failure of an extruded aluminium-7% magnesium specimen fatigued after ageing 15 days at 150° C.

Fig. 10.—Photomicrograph showing much more widespread deformation near failure in an aluminium-7% magnesium specimen fatigued in the solution treated condition.

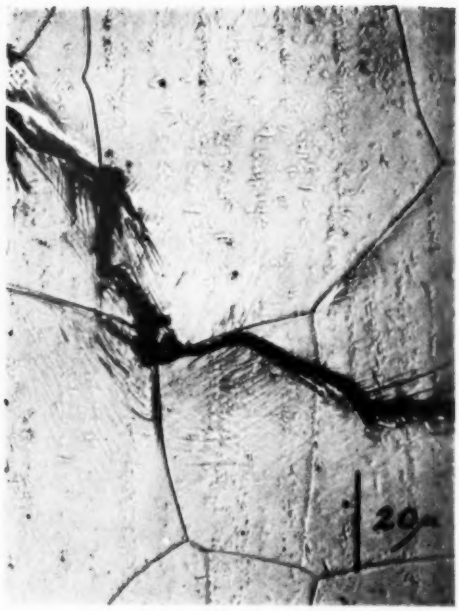

Fig. 11.—Photomicrograph showing the transcrystalline nature of the greater part of the fracture on an aluminium -7% magnesium specimen fatigued in solution treated and aged 15 days at 150° C. condition.

Examination of Rolls Royce Specimen Fractures.

Examination of the fracture surfaces showed that in the extruded material failure was transcrystalline on specimens fatigued in the as-extruded condition, but that ageing this material for various periods at 150°C. did produce a small amount of intercrystalline fracture, although in every case and at all stress levels the failure was predominantly transcrystalline. After solution treatment, a few intercrystalline facets were visible on specimens fatigued in all conditions of heat treatment, including the solution treated condition, although, as in the case of extruded material, the fractures were mainly transcrystalline.

Metallographic Observations on the Relation between Fatigue Behaviour and Microstructure.

Metallographic specimens were machined from the extruded strip and individual specimens fatigued at room temperature in similar conditions of heat treatment to those for which S-N curves had been determined. Specimens were electropolished after heat treatment.

Fig. 8 shows the deformation produced on the as-extruded specimen. It can be seen that transcrystalline failure has occurred with relatively localised deformation. A small amount of intercrystalline fracture was observed on specimens fatigued after ageing for 1 day at 150°C. although the failure was still predominantly transcrystalline. When aged for 15 days at 150°C., the failure was still predominantly transcrystalline, although again a few examples of intercrystalline failure were observed, as shown in Fig. 9. Specimens were then solution treated at 420° C., quenched into cold water, and individual specimens tested in the solution treated condition and after ageing for various periods at 150°C. Fig. 10 shows the deformation and type of fracture produced on specimens fatigued in the solution treated condition. It can be seen that the deformation was much more widespread than that produced on the as-extruded material. After solution treatment and ageing for 1 day at 150°C., a small amount of intercrystalline cracking was observed, although again the failure was mainly transcrystalline. Fig. 11 shows the transcrystalline nature of the majority of the fracture produced on specimens solution treated and aged 15 days at 150° C., a condition in which precipitation was present both at the grain boundaries and within the grains. Thus, the metallographic observations agree with those made during the examination of Rolls-Royce specimen fractures.

TABLE I.—MECHANICAL PROPERTIES OF ALUMINIUM-7% MAGNESIUM ALLOY IN VARIOUS CONDITIONS OF HEAT TREATMENT.

Condition	Limit of Proportionality (tons/sq. in.)	0·1% Proof Stress (tons/sq. in.)	0·5% Proof Stress (tons/sq. in.)	Ultimate Tensile Stress (tons/sq. in.)
As extruded	8·3	9·2	9·35	20·0
Extruded + 2·4 hours at 150°C.	8·9	9·5	9·8	20·2
Extruded + 1 day at 150°C.	8·7	9·5	—	20·2
Extruded + 15 days at 150°C.	7·8	8·4	9·3	20·5
Solution treated	5·1	7·1	8·5	18·5
Solution treated + 2·4 hours at 150°C.	5·4	7·1	8·0	17·9
Solution treated + 1 day at 150°C.	5·4	6·9	8·1	18·0
Solution treated + 15 days at 150°C.	6·7	8·4	10·1	21·4
Solution treated, cold rolled 75% reduction	16·5	27·4	29·2	30·3

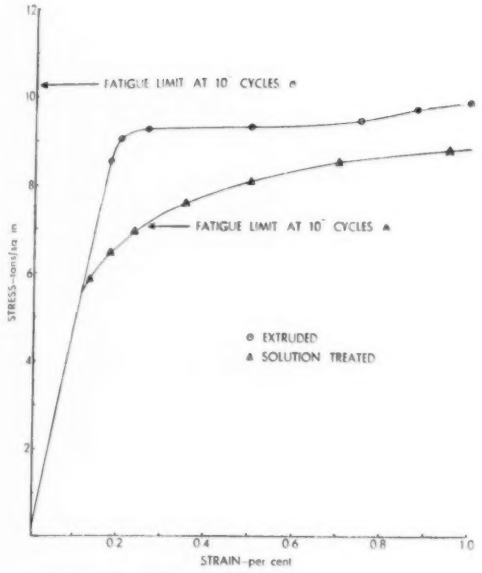

Fig. 12. Stress strain curves for aluminium-7% magnesium in the as-extruded condition and after solution treatment at 420° C. for 16 hours and cold water quenching.

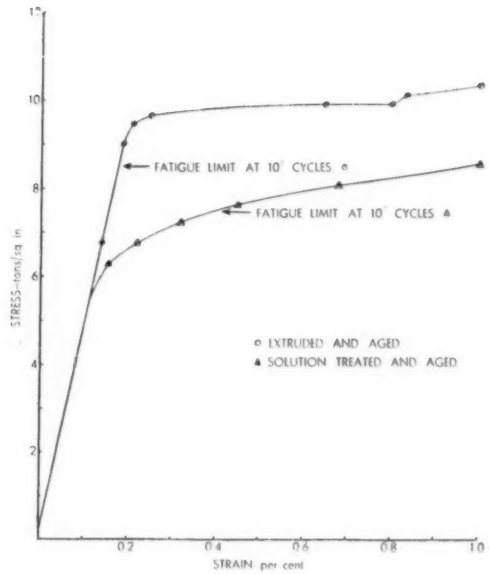
FATIGUE LIMIT AT 10⁷ CYCLES ○
FATIGUE LIMIT AT 10⁷ CYCLES ▲
○ EXTRUDED AND AGED
▲ SOLUTION TREATED AND AGED
STRESS—tons/sq in
STRAIN—per cent

Fig. 13. Stress-strain curves for aluminium-7% magnesium aged 1 day at 150° C. after extrusion and after solution treatment at 420° C. for 16 hours, followed by quenching in cold water.

Tensile Tests

Tensile tests were carried out on the alloy in similar conditions of heat treatment to those for which S-N curves had been determined. The tensile properties are shown in Table I. Stress-strain curves for the as-extruded and solution treated conditions are shown in Fig. 12. It can be seen that in the as-extruded condition a marked yield point is present, but that the solution treated material exhibits plastic behaviour at a much lower stress with a more gradual departure from Hooke's Law. After ageing for 1 day at 150°C., (Fig. 13), the yield point on extruded material is still very much in evidence, although slightly less marked than in the as-extruded condition. The shape of the stress strain curve for solution treated and aged material does not alter apprec-

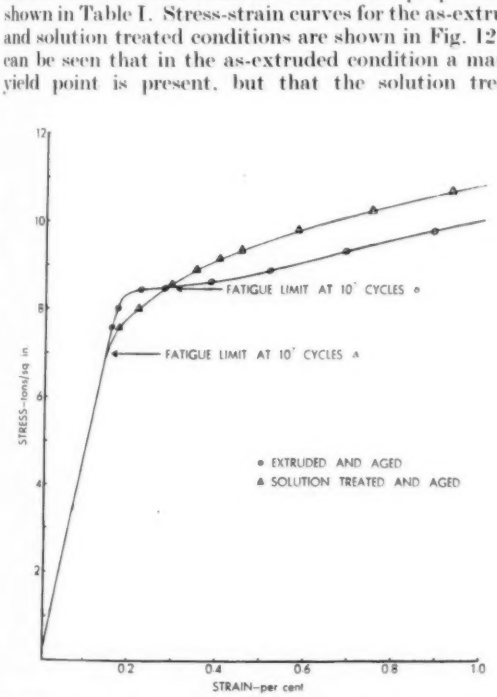
FATIGUE LIMIT AT 10⁷ CYCLES ○
FATIGUE LIMIT AT 10⁷ CYCLES ▲
○ EXTRUDED AND AGED
▲ SOLUTION TREATED AND AGED
STRESS—tons/sq in
STRAIN—per cent

Fig. 14.—Stress-strain curves for aluminium—7% magnesium aged 15 days at 150° C. after extrusion and after solution treatment at 420° C. for 16 hours, followed by quenching in cold water.

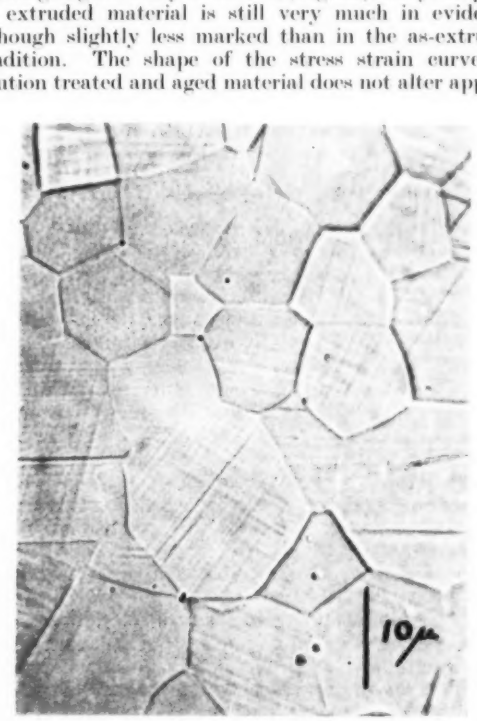

Fig. 15.—Photomicrograph showing extensive fine slip in aluminium-7% magnesium stressed to 9·0 tons/sq. in. in the as-extruded condition.

iably with such treatment. Stress strain curves determined after ageing extruded and solution treated material for 15 days at 150°C. are shown in Fig. 14. After this treatment, the marked yield point in the extruded material has virtually been removed, and it is of interest to note that the stress-strain curve for solution treated and aged material intersects that of the extruded and aged material during the initial yield of the latter, and thereafter the rate of work hardening of the two materials appears to be similar.

A metallographic examination was then made of the tensile deformation produced on material in the solution treated condition. Specimens were machined from the strip extrusion, heat treated, and electropolished prior to testing. Specimens were loaded to various stress levels for 15 sec. and after removal from the tensile machine, examined microscopically. It was found that deformation in the form of isolated slip bands could be detected at a stress as low as 1·5 tons/sq. in., and at 3·0 tons/sq. in. deformation on more than one slip plane within individual grains could be observed. Both these stresses are below the nominal yield point of the material. With increase in stress above 3·0 tons/sq. in., there was a marked increase in the number of slip planes operative. In the extruded condition, it was possible to detect deformation at a stress level well below that at which marked yielding occurred. Deformation was observed after stressing to 6·0 tons/sq. in. With an applied stress of 9·0 tons/sq. in., considerable deformation occurred, with extensive fine slip and two or three slip planes operative within individual grains: this is shown in Fig. 15.

Examination of Tensile Fractures.

The 0·25 in. B.S.F. tensile specimens on which the tensile properties had been determined were examined in all conditions of heat treatment, and in each case ductile transcrystalline failure was observed. The cold worked material exhibited the lowest elongation (8%), but in all other conditions the elongations were of the order of 40%.

Discussion

Examination of the S-N curves shows that the as-extruded condition, which exhibits a marked yield point in the tensile test, has a steel type curve with a knee at 5×10^5 cycles and a fatigue ratio of 0·5. Ageing this material for a short time—2·4 hours at 150°C.—tended to remove the knee on the curve and to produce an S-N curve characteristic of aluminium alloys. The fatigue limit at 10^7 cycles was at the same time reduced by 1·75 tons/sq. in., and as the tensile strength was virtually constant the fatigue ratio fell to 0·42. Ageing for as long as 15 days at 150°C. produced no further significant change in the S-N curve, although a considerable change in the shape of the stress-strain curve occurred, together with changes in the microstructure. It has been shown that ageing the extruded structure for 15 days at 150°C. produced considerable precipitation and virtually removed the yield point.

When the extruded material was solution treated to produce an increase in grain size, S-N curves typical of aluminium alloys were obtained in the solution treated condition an in all aged conditions. It can be seen from Fig. 6 that the fatigue properties are almost constant for all conditions, with a tendency for the best fatigue properties to occur after ageing for 1 day at 150°C. Ageing for 15 days at 150°C. produced a slight increase in the tensile properties and the fatigue ratio therefore fell from 0·36 in the solution treated condition to 0·32 when fully aged. These observations suggest that small grain size has a beneficial effect on the fatigue properties of this alloy.

Levy[2], discussing the influence of strain ageing on fatigue life, concludes that strain ageing offers evidence of both a qualitative and quantitative nature to account for the existence of a sharp knee in the S-N curves of materials which readily strain age under tensile conditions. He suggests, however, that ability to strain age in a tensile test is not a necessary condition for strain ageing to occur in a fatigue test, as cyclic stress might induce metallurgical changes which would allow strain ageing to occur during such a test. A knee might then be observed on an S-N curve, though there may be no indication of a sharp yield point in the conventional tensile stress-strain curve. The present observations indicate that the converse of this is also true, the extruded alloy aged 1 day at 150°C. having a well defined yield point on the stress strain curve, but having an S-N curve without a sharp knee.

Results published by H. F. Moore[3], for 0·93% C steel fatigued after different heat treatments, show that the position of the knee in the S-N curve is dependent on the previous thermal treatment of the material. In the annealed and sorbitic conditions, the knee occurred at 2×10^6 cycles and 5×10^5 cycles, respectively, and there was complete absence of fracture points between a few million cycles and one hundred million cycles. In the troostitic condition, however, the knee occurred at 10^5 cycles and fracture points were obtained for all values of N. Examination of the S-N curve obtained for aluminium-7% magnesium in the as-extruded condition shows that it is similar to that obtained for troostitic steel, both in the early knee position and the fracture points for all values of N.

In their description of the yield point phenomena and stretcher strain markings in aluminium-magnesium alloys, Phillips, Swain and Eborall[4] refer to the large initial yield observed on the stress-strain curves of these alloys in suitable conditions as Type A yield. The many subsequent yields which occur are designated Type B. The authors attribute the Type A yield point to the blocking of the propagation of slip from grain to grain, owing to the presence of a high concentration of magnesium at the grain boundaries. The observations during the present investigation show that well defined Type A yielding occurs in conditions which show marked grain boundary precipitation, and when in fact the grain boundaries have reached a stage beyond that at which segregation of magnesium atoms might be said to have occurred.

The metallographic examination and study of the fatigue fractures obtained on Rolls Royce specimens showed occasional examples of partial intercrystalline fracture in most conditions of heat treatment. This demonstrates that fatigue failure can occur through the boundaries in conditions in which a completely transcrystalline tensile fracture would be obtained. Earlier observations on a ternary aluminium alloy containing 7·5% zinc and 2·5% magnesium have shown that the converse of this is also true, and that completely transcrystalline fatigue failure can be obtained on material having a brittle intergranular tensile fracture. Solution treatment of the ternary alloy followed by ageing for 2·4 hours at 150°C to maximum tensile strength produced

grain boundaries depleted of solute atoms. Intercrystalline fracture occurred if tensile tests were carried out on such material. Fatigue tests were also carried out in this condition and examination of the fractures of Rolls Royce specimens revealed that the amount of intercrystalline fracture was greatest at high fatigue stress. At all stresses, including high stress, the initiation of the failure was predominantly transcrystalline, although there was some evidence of grain boundary initiation. When grain boundary initiation did occur, however, the fracture very rapidly propagated in a transcrystalline manner. Intercrystalline propagation occurred if a depleted grain boundary was suitably aligned with respect to a developing transcrystalline crack and the observation of a few intercrystalline facets on the fatigue fractures of the aluminium-7% magnesium alloy are probably due to this mechanism. It is suggested, however, that most of the intercrystalline portion of the fracture observed on the fatigue specimens of the aluminium-zinc-magnesium alloy is the tensile failure which occurs when the section of the specimen has been sufficiently reduced by the transcrystalline fatigue crack. Thus, it would appear that the application of cyclic stress to these alloys produces soft spots within the matrix along which crack propagation proceeds, and which are preferred by the developing fatigue crack to the pre-existing soft spots at the grain boundaries produced by heat treatment.

In the cold worked condition, the S-N curve was typical of those obtained on aluminium alloys, but the fatigue limit at 10^7 cycles was 5·0 tons/sq. in. higher than that obtained in the solution treated condition. The fatigue ratio increased from 0·36 in the solution treated condition to 0·38 when cold worked. Cold working therefore has a beneficial effect on the fatigue properties of solution treated aluminium-7% magnesium.

Conclusions

(1) Grain size is important in determining the fatigue properties of aluminium-7% magnesium.

(2) It would appear that only the as-extruded condition of aluminium-7% magnesium produces the steel type S-N curve: as soon as any heat treatment is applied, whether ageing or solution treatment, the S-N curves obtained are typical of those obtained on other aluminium alloys.

(3) Cold working appears to have a beneficial effect on the fatigue properties of solution treated aluminium -7% magnesium and gives a relatively high fatigue ratio, although not as high as the extruded material.

(4) Materials subjected to cyclic stress can exhibit partial intercrystalline fracture in conditions in which, if the stress were unidirectional, the failure would be entirely transcrystalline. Transcrystalline fatigue failure has been observed on materials whose tensile fracture is intercrystalline.

(5) Well-defined yield points can be observed on aluminium-7% magnesium of suitable grain size in conditions which show considerable precipitation at the grain boundaries.

Acknowledgments

The author is indebted to Mr. P. J. E. Forsyth for helpful discussions and to Mr. N. J. F. Gunn for the tensile testing of the alloy.

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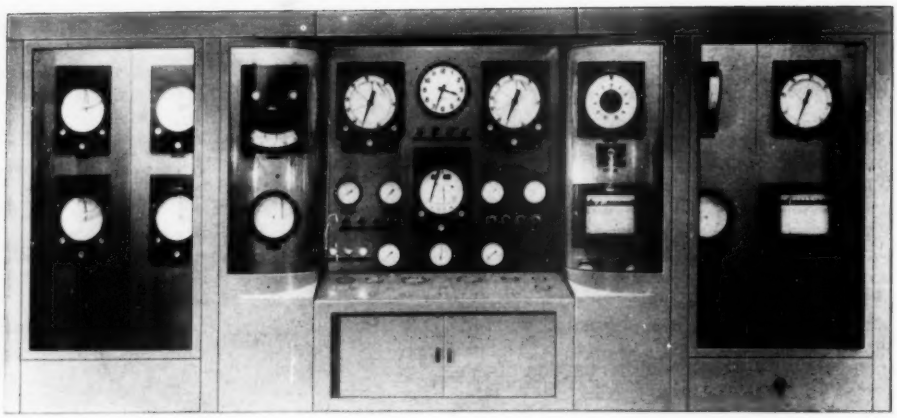
Control System Sets Own End Point

AT Colvilles' new Ravenscraig Works, the first-hand melters in charge of open-hearth furnaces have a new ally—an automatic control system which integrates the control of five variables. Constant optimum conditions maintained by this system enable oil- and gas-fired open-hearth furnaces to produce better steel for less fuel, lengthen refractory life, and eliminate hold-ups due to roof-melts. The installation, designed and manufactured by Honeywell Controls, Ltd., provides safe high temperature operation, and automatic action when critical variables reach peak values. Among the eventualities which the control system copes with automatically are shortage of either fuel, excessive furnace roof temperatures, and the periodic need for reversal of firing.

The system exploits to the full the potentialities of cascade control—a method of instrumentation in which the set-point of a controller for one variable is determined by another master controller which controls a second related variable. Cascade control provides closer control of conditions where many variables are involved, and increases the number of operations which can be carried out automatically. Indexet mechanisms—the essential link in cascade control systems—reset a secondary controller in accordance with the requirements of a master controller. A varying pneumatic signal from the master controller is received by an Indexet mechanism fitted to the secondary controller. Acting in proportion to the pneumatic signal, the Indexet resets the secondary controller over any desired range.

On the furnaces at Colvilles', final control action is taken by separate flow controllers for oil, gas, steam and combustion air flow. The settings on these controllers are themselves determined by a total fuel controller, which continuously measures total fuel input, and compensates for any lack of either fuel by raising the set-point on the controller for the other fuel. Total B.Th.U. supply thus remains independent of random variations in oil or gas supply.

The total fuel supply is in turn subject to the overriding action of two roof temperature controllers, acting on signals from Radiamatic radiation pyrometers focussed on each end of the roof. When either of these temperatures approaches danger-point, one of the highly



General view of the panel.

sensitive roof temperature controllers takes over, and holds the furnace temperature accurately at peak operating value. During the final stages of the melt, the two roof temperature controllers are adjusted in tandem to give closer regulation of heat-input.

Manual or automatic reversal is selected by a switch. Automatic reversal occurs, either on a time-signal or on high-temperature signals from Radiamatics sighted on the North and South checkers. Oil, gas and steam valves then close in the right sequence, the dampers change over, and the fuel and air are re-admitted correctly. Integrated furnace pressure control assists firing conditions, and maintains a slight positive pressure throughout the melt, preventing heat loss by ingress of cold air.

The instrument panel is designed to have a central operating section giving a bold indication of variables, with glass-enclosed side panels for protection of control and recording instruments. Checking facilities are provided at the rear of the panel, together with fuses, filters and auxiliary equipment.

Conference on Forge Modernization

A two-day conference arranged by the British Iron and Steel Research Association on the subject of "Modernization of Forges" was held on the 16th and 17th of September, at Ashorne Hill, near Leamington Spa, under the Chairmanship of Mr. F. J. Somers of Walter Somers, Ltd., Halesowen. More than forty member firms of the Association were represented by more than a hundred participants in the conference.

Three papers were presented for discussion. The first, entitled "Building a New Forge" was given by Mr. A. C. Lowe of Thos. Firth & John Brown, Ltd., who spoke about the rebuilding of the 1,750 ton press shop at their Atlas Works. He described the factors which had to be taken into account when the question of rebuilding was under consideration, and the way in which their new plant was fitted-in with the existing facilities. Mr. Lowe concluded by giving their present thoughts regarding the layout and operation after some months' working experience of them.

The second paper, dealing with features that make for a well-designed modern forge, was given by Mr. E. Homer Kendall, of Kendall Contracting, Inc., Alliance, Ohio. He stressed the need to allocate separate space to (*i*) the heating process, (*ii*) the forging process proper, and (*iii*) the laying down of completed jobs and any necessary heat treatment, so as to ensure the greatest flexibility in operation and unimpeded flow of work. He then went on to discuss the factors which determine the type of equipment which should be installed in each. His paper contained many references to forge layouts with which he was acquainted which illustrated the points he was making.

The last paper, given by Mr. J. Banbury of the B.I.S.R.A. Operational Research Department, was entitled "A Method for Comparing Alternative Ways of Forging." Mr. Banbury, referring to work carried out by his Department, showed how the method had been used by a company to compare an existing forge with a proposed new forge for the purpose of deciding whether to re-equip. He also described the way in which the same technique could be used for comparing different forges so that cost and time-saving factors could be pin-pointed.

The discussion on these papers was extremely lively, indicating the wide interest in the forging industry today in modernization. Latest developments excited particular interest, and were further emphasized by the showing of a film of the B.I.S.R.A. Experimental Forge, in which the development of automatic forging is at an advanced stage. A further film by The English Steel Corporation, bore witness to the wide range of processes and equipment required in forging.

After the formal conference session, the members visited the forges of Walter Somers, Ltd., Halesowen, by courtesy of the company, and there saw in operation a new light forge of advanced design.

The Development of Beryllium for Nuclear Reactor Applications

By G. C. Ellis, B.Sc., M.Sc., F.I.M.

Atomic Weapons Research Establishment, Aldermaston

There is a considerable interest at present in the use of beryllium as a canning material in nuclear reactors, for which purpose it has attractive nuclear and high temperature properties. Its use in this field presents formidable development problems due to the difficulties encountered at various stages in the chemical and metallurgical processing of the metal. In the first part of this article the author discusses the demand for beryllium in this field, toxic hazards and the initial production of the metal from the ore. Subsequent parts will deal with the production of ingot and powder metal, fabrication, mechanical properties, and the possibility of improving the ductility.

BERYLLIUM is a metal possessing very attractive nuclear and high temperature properties, which have many potential reactor applications; yet, it may be regarded, to date, as the Cinderella metal of atomic energy. Only restricted reactor use in low stress applications has been achieved so far (in the U.S.A. and the U.S.S.R.). This is attributable to the many formidable difficulties encountered throughout the chemical and metallurgical processing of the metal, from its initial mining to its final fabrication. Of these, the main limitation has always been the relatively low ductility of the metal, leading to difficulties of fabrication and joining, and resulting in inadequate properties for higher stress applications. There are two possible solutions to this problem of inadequate ductility: these are discussed in detail later.

At present, there is a greatly increased demand for beryllium, largely for reactor purposes, both in the U.S.A. and the U.K. The author, at the Atomic Weapons Research Establishment, Aldermaston, has been connected with the Industrial Group of the Atomic Energy Authority in the development of beryllium for gas-cooled reactor applications. In the present article, the reactor demand for beryllium is discussed, and then an outline is given of the difficulties encountered and the achievements made in the extraction, production, fabrication, and property evaluation of beryllium for reactor applications. Any opinions expressed are those of the author, and do not necessarily agree with the official attitude of the U.K. Atomic Energy Authority.

The Reactor Demand

With the advent of the exploitation of atomic energy in 1941, American interest in beryllium was greatly increased. Prior to this, although the commercial extraction of the oxide had been established in the 1920's for its ceramic use and for precipitation-hardening alloy production, the production of the metal had been negligible, due to its relative brittleness. In the 1940's an extensive investigation of metal production, fabrication and property evaluation was initiated via the ingot route, but owing to inconsistency of production quality, difficulties of fabrication, and inadequacy of ductility, the project was discontinued. Instead, the commercial production of large metal powder blocks (60 × 24 × 6 in.) suitable for moderator and reflector applications was begun. Even so, only 2–5% of the beryl ore consumed in the U.S.A. was used for metal production, compared with 85% for copper-beryllium alloy production and some 10% for beryllium oxide production.

Within the last two years, interest in beryllium for reactors has increased considerably in both the U.S.A. and the U.K. The U.S. Atomic Energy Commission has ordered the production over the next five years of 500 tons of reactor grade metal by the Beryllium Corporation, Reading, and the Brush Beryllium Co., Cleveland, at an average cost of $47 lb. The form of the metal (ingot or powder) and its intended reactor (or other) uses are not disclosed. In the U.K., beryllium is under development by the Atomic Energy Authority and by the industrial consortia for use in advanced, gas-cooled, graphite-moderated, power reactors of the Calder Hall type. Similar development is believed to be in progress in France, and possibly in other countries.

This increased demand for beryllium is due to the fact that the very attractive properties of the metal as a potential nuclear engineering material can no longer be ignored by those responsible for reactor development. In the U.K. the first stage of the atomic power production programme is based on the development of the gas-cooled, graphite-moderated reactor. The use of magnesium alloy fuel cans in the present Calder Hall reactors restricts the maximum fuel surface temperature to the region of 400° C. Higher operational efficiency necessitates the use of higher fuel temperatures for improved heat transfer, and for this purpose, beryllium offers good high temperature resistance and excellent nuclear properties.

For nuclear use, beryllium possesses high neutron scatter and low neutron capture cross-sections; the latter, being 1/20th that of zirconium, is the lowest value exhibited by any metal. It also has high efficiency as a neutron source on alpha particle irradiation. In addition, for high temperature use (reactor or otherwise) beryllium usefully combines low density (1·85 g./cc.), high thermal conductivity (40% that of copper), high melting point (1,285° C.), high elastic modulus (40 × 10^6 lb./sq. in.) and good water corrosion resistance, with atmospheric corrosion resistance and strength-weight ratio (up to 500° C.) superior to those of titanium.

On the basis of these properties, beryllium is clearly suited for the following reactor applications :

neutron moderator, neutron reflector	as large machined blocks
fuel can	as welded tube or sheet
structural component ..	as welded rod or sheet

Moderator and reflector applications require only sufficient ductility to withstand the thermal stresses imposed during reactor service. This has been achieved in the U.S.A., where beryllium was used as a moderator in the Submarine Intermediate Reactor, and as a reflector in the Materials Testing Reactor ;[2] both applications involved the use of vacuum hot-pressed metal powder blocks in the machined condition. It is understood that a beryllium moderator similarly produced has been used in the U.S.S.R.[3]. In Russia and in America, fuel canning and structural component applications of beryllium do not appear to have been achieved, due presumably to difficulties associated with the inadequate ductility of the metal. It is of particular interest, therefore, that beryllium is at present under development in the U.K. for canning and structural component applications. In the author's opinion, the solution to the canning problem will be resolved by the development of maximum properties (by the use of improved metal quality and improved fabrication techniques) combined with a relaxation of the requirements of the design engineers (by the suitable choice of fuel and a modification of can design).

It is conceivable that the establishment of adequate production capacity and the development of suitable fabrication techniques and mechanical properties for reactor use may lead to elevated temperature applications of the metal outside the atomic energy field, where strength/weight ratio is a primary consideration.

Development Difficulties

The many development difficulties which occur throughout the chemical extraction and the metallurgical processing of beryllium appear to have two distinct origins. These difficulties are discussed individually in the subsequent sections, but first, a brief survey of them will be given.

In the first place, there are problems attributable to the chemical and physical properties of the metal and its compounds ; these are the more numerous but not the more important of the development difficulties encountered. The notable properties include the chemical inertness of beryl ; the stability of the oxide with respect to normal reductants ; the hygroscopic nature of and the reactivity of the beryllium halides ; the high reactivity and melting point of the metal with respect to production via the ingot or powder routes ; and the toxicity of the metal. These properties give rise to extraction and production problems, and are responsible for the important lack of complete purity in commercial quality beryllium, and to a certain extent for the high cost and the low volume of production of the metal. Chemical and physical properties are also responsible for some of the difficulties encountered in the fabrication, machining and joining of beryllium. As a hexagonal metal, beryllium is highly susceptible to preferred grain orientation on fabrication. Fabrication difficulties are also caused by the tendency of beryllium to oxidise at elevated temperatures and to pronounced seizure and galling on die tools. The abrasive nature of the metal causes excessive tool wear on machining, while oxidation is also partly responsible for the difficulty in metal joining by welding or brazing. These difficulties are not insuperable. They can be overcome by the use of special techniques, which, unfortunately, further increase the cost of the metal.

The second, and by far the more important, type of difficulty has already been mentioned, and is attributable to the inadequate ductility which is encountered in fabrication and joining, which severely limits the usefulness of the metal. Preferred grain orientation, together with limited modes of deformation, give rise to a pronounced property anisotropy of the fabricated metal with relatively poor transverse ductility. Inadequate ductility precludes final cold operations for improved surface finish and dimensional tolerances. The major problem in welding is that of crack formation, due to hot shortness and cold brittleness. For canning purposes, a main requirement is high creep ductility in all directions in the fabricated metal. This is difficult to attain.

The ductility problem of beryllium may be approached in two ways. The best properties of the commercial grade metal may be developed by the fabrication control of orientation and structure so as to minimise property anisotropy. This may permit the realisation of acceptable two-dimensional ductility values, but possibly at the expense of a complete lack of ductility in the third dimension. This may not be important. The joining difficulty would remain, however, and would require the development of special techniques. The alternative approach to the problem lies in the preparation of pure beryllium (a very formidable task) for the realisation of its inherent properties. This is based on the hypothesis, which is not universally shared, that the relative brittleness of the commercial metal is due to the presence of soluble impurity atoms. The successful preparation of pure ductile beryllium would relieve the fabrication and joining difficulties and provide metal of acceptable properties for reactor use. It is doubtful, however, whether this degree of purity could be achieved, particularly in large scale production.

Toxic Hazards

There has been much controversy, especially in the immediate post-war period, over the toxic hazards of beryllium and its compounds. Two main facts have emerged from recent experience. One is that although the metal and its compounds are toxic, provided the recommended air tolerances are observed, there is little or no hazard involved with these materials. The other is that the recommended air tolerances are readily attained without great inconvenience.[4]

In human beings, beryllium and most of its compounds can cause skin and lung diseases. On unbroken skin it can cause dermatitis, but this appears to be largely dependent on individual susceptibility. Tumours and ulcers are caused by beryllium and its compounds entering cuts and abrasions, but these are localised and are not serious. On inhalation of airborne particles, two diseases of the lung may occur, both of which can be fatal. Chemical pneumonia may be caused as a result of a brief but high exposure, while berylliosis normally results from long exposure to mild doses. An unusual factor is that the latter can be fatal even after latent periods of up to 11 years' duration. Both types of lung disease appear to be dependent on individual susceptibility, which also is most unusual. As there is no known method of predetermining susceptibility in individuals,

all persons exposed to beryllium must be protected. It is interesting that there appears to be no recorded case of beryllium poisoning due to ingestion. In fact, it is rather amusing to recall that the original name for beryllium was glucinium, due to its sweet taste.

The air tolerances recommended for beryllium plants by the U.S. Atomic Energy Commission in 1949, which are still accepted internationally are :

(1) for in-plant conditions, the daily 8-hour average must not exceed 2μg. cu. m. of air, and for no period (however short) must the peak value exceed 25 μg. cu. m.

(2) for out-plant conditions, the monthly average must not exceed 0·01 μg. cu. m.

These tolerances are achieved by careful plant design combined with good ventilation and air cleaning systems. These engineering precautions must be supplemented by adequate staff instruction and hygiene, and by the good housekeeping control of ingoing and outgoing material.[5] It is well to remember that an unintelligent or badly informed workman can nullify the results of good design and ventilation.

The overall efficiency of control is determined by regular air sampling inside and outside the plant, and by the smear testing of exposed in-plant surfaces. The toxic hazards and their control increase production difficulties and cost.

Ore Supply

Beryllium occurs in about thirty minerals, but the one ore, to date, is beryl—beryllium aluminium silicate (3 BeO. Al_2O_3. 6 SiO_2). Commercial grades used at present contain 10–12% beryllium oxide, i.e. 3–7% beryllium metal. The main sources of beryl are Brazil, South Africa, Rhodesia, the Argentine, Morocco and Madagascar.

Part of the high cost of beryllium is due to the high cost of beryl. This, in turn, is due to the small mining volume and the low recovery efficiency (about 30%) by the present hand picking methods. It appears that the supply of present quality beryl would accommodate a substantial increase in the total industrial demand[2]. A great increase in future demand, however, would necessitate the development of mechanical concentration methods for the recovery of fine, low grade deposits. Much work seems to be in progress on the beneficiation of these deposits by flotation and other methods. Since the world resources amount at present to a few hundred thousand tons of beryllium metal, it seems unlikely that beryllium will ever become a major tonnage metal.

Extraction of the Oxide

The extraction of beryllium oxide from beryl is difficult and expensive, due to the inertness of beryl and to the presence in it of aluminium, which is chemically similar to beryllium. In the U.S.A. two processes are operated commercially.[4] At the Beryllium Corporation, beryl is converted by high temperature roasting to the soluble fluoride, which is separated from the insoluble impurities by leaching. The addition of caustic soda precipitates the hydroxide, which is then calcined to the oxide. At the Brush Beryllium Co., beryl is melted at 1,700° C. and is then quenched in water and heat treated, when it becomes reactive to sulphuric acid. The soluble beryllium and aluminium sulphates are removed by leaching. Most of the aluminium is precipitated as alum on the addition of ammonium hydroxide. Sequestrants are added to retain impurities in solution, and caustic soda is added to form the aluminate and beryllate. On boiling, the beryllate decomposes to the hydroxide, which is finally calcined to the oxide.

The oxide produced by the fluoride process in the U.S.A. is reputed to be less expensive and less pure than that produced by the sulphate process. It is used mainly for beryllium alloy production. The sulphate product contains 0·05% iron, 0·05% aluminium, 0·05% silicon and 0·15% sodium. For use as oxide, the sulphate product is further purified with respect to sodium and aluminium.

The extraction processes used in the U.S.S.R. and in France are not known. In the U.S.S.R., as in the U.S.A., the technical grade oxide used for metal production is further purified before use as oxide for moderator purposes.[3] In the U.K. a modified fluoride process is operated by the Atomic Energy Authority at its Milford Haven plant,[7] where relatively high purity oxide is obtained.

It would seem that the first logical step in the development of high grade commercial quality metal is the production of beryllium oxide of maximum possible purity ; further purification should then be possible in the extraction of the metal from the oxide. This should be combined with careful production control to minimise (or eliminate) contamination from material and atmospheric sources. It is always easier to eliminate impurities before, rather than after, the ingot stage in metal production processes, and beryllium production is no exception to this general rule.

Reduction of the Oxide

Beryllium oxide has a high heat of formation (141 k. cal. mol.), and cannot be reduced completely to the metal. The beryllium halides are more reducible than the oxide, but they are expensive, reactive and hygroscopic—particularly the chloride.

The beryllium halides cannot be reduced by hydrogen. Reduction by alkali or alkaline earth metals is possible, and this forms the basis of one commercially operated process, via the fluoride. The low boiling point of the chloride (490° C.) restricts metal reduction methods to low temperature vapour reactions or to high temperature bomb reactions, neither of which is attractive for commercial operation. The electrolytic reduction of the beryllium halides is restricted to the use of fused salt baths, since aqueous solution electrolysis is precluded by the strong affinity of beryllium for oxygen, and while there are certain non-aqueous solvents available such as ammonium hydroxide, they are few and impractical.[8] In fused salt electrolysis, the use of the chloride is to be preferred because of its lower melting point (419° C.), compared with that of the fluoride (800° C.). This constitutes the second commercial process for metal production.

Most of the beryllium is produced in the U.S.A. by the Brush Beryllium Co., and the Beryllium Corporation, both of which firms operate the thermal reduction process. This consists of the conversion of the oxide to ammonium beryllium fluoride, the thermal reduction of the double fluoride at 900° C. in a graphite crucible, and the subsequent reduction of the beryllium fluoride by 70% stoichiometric magnesium in a graphite crucible at a maximum temperature of 1,300° C. After washing, the resulting pebble metal contains 97% beryllium,

Fig. 1.—Available routes for the conversion of pebble and flake beryllium metals to the ingot and powder forms.

0·2–0·5% metallic impurities, around 0·2% oxygen, and 2·0–2·5% magnesium plus magnesium fluoride. The metal is produced in 30-lb. batches. Approximately 62% is recovered as pebbles, whilst some 34% is recycled as excess soluble beryllium fluoride. The two main disadvantages of the process appear to be the difficulty experienced in obtaining an efficient separation of slag and metal, and the high temperature contamination of the melt.

In France, the electrolytic reduction process is operated by the Pechiney Co. No information has been published on the details of the method used. This process was also operated on a semi-commercial basis by one firm in the U.S.A. The sequence of operations in the electrolytic process consists of the chlorination of the oxide with carbon at 800° C.; the purification of the chloride by distillation at 500° C.; and the electrolysis at 400° C. of a beryllium chloride : sodium chloride, 50 : 50 eutectic mixture, using a graphite anode and a nickel cathode container. The resulting flake metal after washing contains 99·7–99·8% beryllium, less than 0·05% of any single metallic impurity, 0·1% oxygen and about 0·1% chloride. The efficiency of the process is reputed to be lower than that of the thermal reduction process, but the resulting metal purity is much higher. The Brush Co. claims that the electrolytic process does not compete technically or economically with the thermal reduction route, particularly for large scale operation.

Both production processes are used in the U.S.S.R.,[3] and are under development by the Atomic Energy Authority in the U.K. Bryant[7] has published information on the electrolytic reduction of the chloride as carried out by Murex Ltd. at Milford Haven, while Derham[8] has given an account of the magnesium thermal reduction of the fluoride as operated by the Imperial Smelting Corporation at Avonmouth.

Consolidated powder metals produced direct from comminuted pebble and flake beryllium exhibit inferior corrosion resistance and mechanical properties, due to their residual halide content. These, particularly the chloride, also markedly increase the neutron capture cross-section of beryllium. Thus, the relative merits of the thermal reduction and electrolytic processes depend not only on their respective operational efficiencies, but also on two other factors :

(1) the economy and efficiency obtainable in the purification and the consolidation of the pebble and flake metals to the usable ingot or powder forms of beryllium, and

(2) the relative merits of ingot and powder beryllium with respect to fabrication, joining and the mechanical properties of the fabricated metal.

Until these two factors are fully assessed, a valid estimation of the relative merits of the thermal reduction and electrolytic processes cannot be made.

Fig. 1 shows the available routes for the conversion of pebble and flake beryllium metals to the ingot and powder forms. Since magnesium fluoride is insoluble in water and acid and is non-volatile (M.P. 1,396° C.), economic powder production by the direct milling of pebble metal with fluoride removal by sublimation or leaching is not possible. Vacuum melting to the ingot appears to be necessary for the removal of the fluoride content. Should the ingot metal prove unacceptable on a metallurgical basis, it would be necessary to incur further production cost in the conversion of the ingot to the more acceptable powder form of the metal. This is the commercial process operated in the U.S.A., whereby powder is produced from vacuum melted pebble metal ingot ; on the other hand the production of powder from electrolytic flake ingot metal is the process operated in the U.S.S.R., and by the Pechiney Co. in France. With electrolytic flake beryllium, it is necessary to establish whether the removal of chloride by vacuum sublimation or by acid leaching can be effected for economic powder production by the direct milling of flake.

Kaufmann[9] states that vacuum sublimation is ineffective for the removal of chloride, which seems to be contrary to Russian experience. Williams[10] of A.E.R.E. has reported the removal of chloride from milled flake by acid leaching. The vacuum sublimation method may be preferable for the consistent reduction of the chloride content to its lower limits, without incurring an increase in oxide content and the possible solution loss of beryllium metal. The usefulness of vacuum sublimation is thus important and should be clarified.

For a complete appraisal of the thermal reduction and the electrolytic processes, it seems necessary, therefore, to establish the relative merits of the resulting ingot and powder beryllium with respect to the reactor requirements of fabrication and properties. From the economic viewpoint, it would appear that although ingot is cheaper than the American powder or the Russian and French powders, it may not be cheaper than powder produced by the direct milling of flake. *(To be continued)*

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Recrystallisation of Tungsten Wires

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From observations of mechanical properties and microstructures, it was found that tungsten wires recrystallise firstly by formation of a columnar structure with good retention of strength and ductility. At higher temperatures, secondary recrystallisation occurs with embrittlement and lowered strength. The practically important temperature for onset of this coarsening is very sensitive to the nature of the starting material and its history. A hypothesis is advanced to explain the phenomena and the kinetics of the processes are considered.

TUNGSTEN wires are commonly used or processed under conditions where recrystallisation can take place to at least some degree, and since, in general, the structural changes consequent upon recrystallisation of this metal lead to embrittlement, the conditions of its occurrence are of great importance. This work[1] seeks to examine the processes involved and to ascertain the influences of variables of treatment on the recrystallisation behaviour.

Experimental

Materials and Working

In most cases, wires were received at a diameter of *ca.* 500 μ* and were drawn to suitable testing sizes under controlled conditions in the laboratory. The starting materials varied from " undoped " (i.e. free from intentional additives) to heavily doped metal, which, as powder, contained up to *ca.* 1% of added impurities.

Drawing was carried out at a speed of 10 m./min., using Aquadag as lubricant, and with drafts of 7½% on diameter. After investigating the effect of drawing temperature (see below), the heating boat was held at 580° C. for all subsequent drawing.

Testing

Several mechanical properties were tested for their suitability as a measure of the progress of recrystallisation, but the ultimate tensile strength (U.T.S.) proved to be the most convenient and reproducible.

Heat treatment was carried out in an atmosphere of purified nitrogen-hydrogen (90/10) mixture, after ascertaining that there was no detectable sensitivity to the gases used. Inadequate removal of oxygen and water vapour from the gas led to embrittlement and poor reproducibility of results, presumably due to metal transfer by a cycle mechanism.[2] Heating was effected by the passage through the wires of current from an infinitely variable transformer, measuring the temperature by an optical pyrometer, with the appropriate corrections from brightness temperature. Some tests were carried out with furnace heating to ensure that the manner of heating had no influence upon results.

Microsections were made longitudinally for all specimens and transversely for some. Final polishing was effected electrolytically, followed by etching without removal from the bath, but with a lowered current density. It was established that this etching procedure gave rise to etch pits by development of {100} planes.

* i.e. 500 microns.

Variables Studied

Systematic investigations were made into the influence on the form of the curve of tensile strength plotted against annealing temperature for constant time (except for No. 6) of the following variables :—

(1) Rate of heating
(2) Drawing temperature
(3) Degree of deformation
(4) Specimen size
(5) Interstage annealing
(6) Time of heating
(7) Nature and amount of additives (" dope ")
(8) Surface condition of wire.

Results

A certain degree of similarity in the results on various materials and after differing treatments makes it convenient to discuss immediately the effects of the annealing treatments. As progressively higher temperatures were employed, the tensile strength of the wire fell from its value in the as-worked state. This fall, at first rapid, became more gradual until, at a temperature near 1,400° C., there was little further loss in strength (the value then being about 200 kg./sq. mm.). At higher temperatures, a second stage of softening set in which followed a similar pattern, levelling off near 100 kg./sq. mm.[3]. This behaviour is exemplified in Fig. 1 and others. The strength values referred to were essentially constant for all materials, the shape of the curves being dependent only upon the temperature ranges in which the processes took place.

Associated with the change in mechanical properties, was a progressive modification of the microstructure from the fibrous state through one of columnar grains (at the end of primary recrystallisation) to the final state of a coarsened structure due to secondary recrystallisation and grain growth.

The detailed manner of transition from the initial to the final stages of microstructure varied from sample to sample, but some general comments may be made. In those cases where the coarsening process was not abrupt, and also where the temperature for coarsening was rather low (e.g. below 1,600° C.) it was often observed that the columnar grains appeared to break up into several smaller sections which were approximately equiaxed. In some large grains, small unconsumed

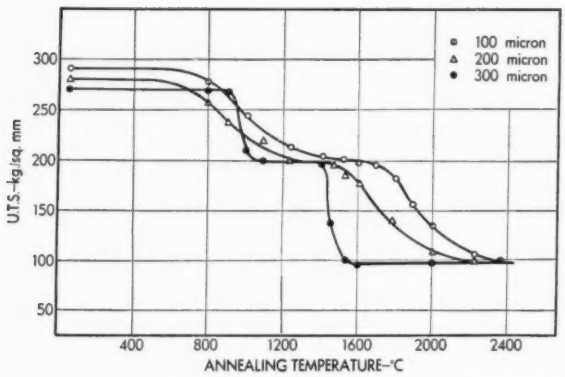

Fig. 1.—Effect on the tensile strength of Sample A of annealing for 10 minutes at various temperatures.

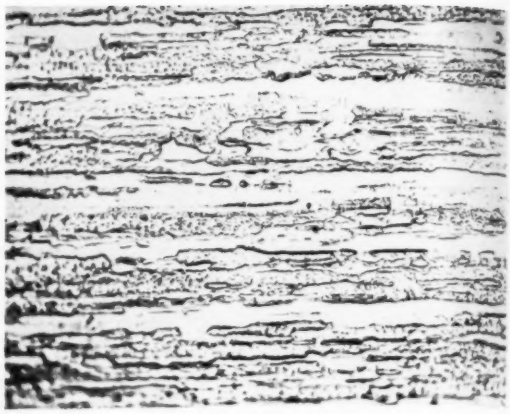

Fig. 2.—Microstructure of 300 micron wire of Sample A, annealed for 10 minutes at 1,425° C. × 1,200

grains, usually elliptical in shape, were seen. These seemingly were absorbed only with difficulty, indicating a low surface energy of the grain boundary between the two grains, due either to a like or possibly to a twin orientation relationship as observed in similar structures reported by Burgers *et al.*[4]. After treatment at higher annealing temperatures, these enclosed grains were absorbed, and grain boundaries generally became smoother and straighter.

Although not always very clearly marked, there was some evidence that grain growth by coalescence started more frequently from the interior than the surface of the wires. This was no doubt due to a small temperature difference resulting from the manner in which heating of the wires was carried out, as, in general, one would expect the recrystallisation temperature to be somewhat lower at the surface where there is greater disturbance of the grains, and the degree of preferred orientation is lower. Similar observations as to the more rapid grain growth at the centre of a wire were made by Jeffries[5] and Nichols,[6] although Johnson[7] refers to growth from nuclei at the surface of lamp wires.

Rate of Heating

Very many workers have found that the rate of heating has a marked effect on recrystallisation, and especially on secondary recrystallisation, and this was examined. Two series of tests were made on the same wire, where one series was raised rapidly (i.e. within a few seconds) to test temperature, and the other slowly with a pause for one minute at 1,000° C. (i.e. within the primary recrystallisation): the two sets of points fell on the same curve and thus betrayed no sensitivity to the change in heating rate; the microstructures were also similar.

Drawing Temperature

There was found to be little influence of drawing temperature on the recrystallisation behaviour of wires drawn at 580°, 700° and 850° C. Drawing at 700° C., however, gave some trouble, the scatter of results being greater than usual. At a heating boat temperature of 700° C., the wire temperature was approximately 600° C.: some discontinuity in property/temperature curves has been reported frequently for tungsten at this temperature.[8-10] All subsequent drawing was carried out at 580° C., to avoid the wire passing through the critical range near 700° C.

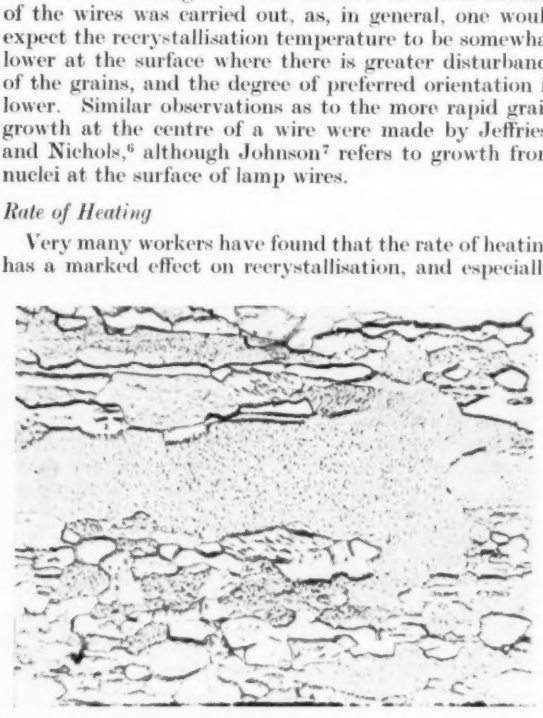

Fig. 3.—Microstructure of 300 micron wire of Sample A, annealed for 10 minutes at 1,450° C. × 1,200

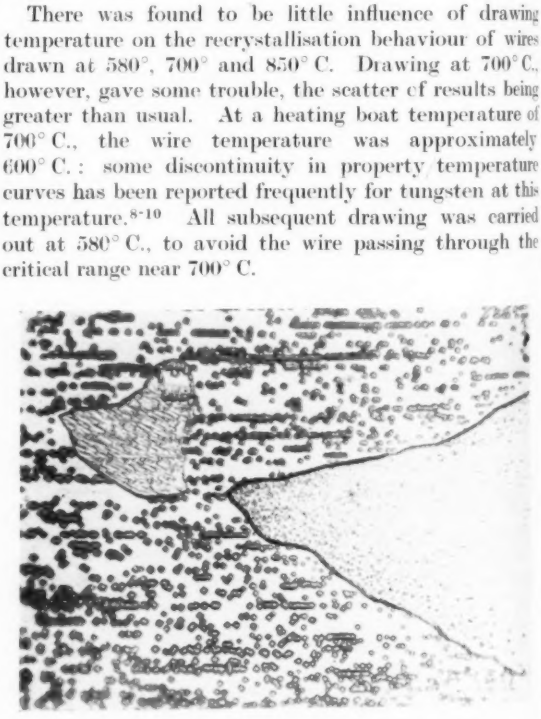

Fig. 4.—Microstructure of 300 micron wire of Sample A, annealed for 10 minutes at 2,000° C. × 1,200

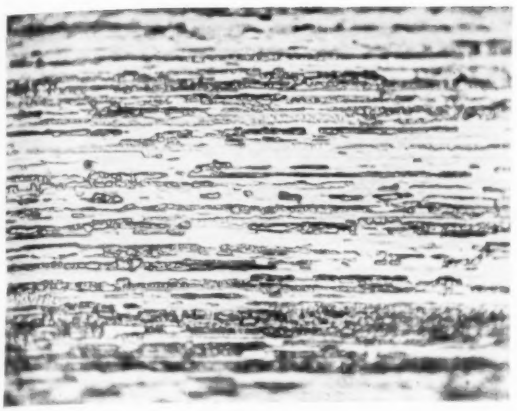

Fig. 5.—Microstructure of 100 micron wire of Sample A, annealed for 10 minutes at 1,800° C. × 1,200

Fig. 6.—Microstructure of 100 micron wire of Sample A, annealed for 10 minutes at 2,100° C. × 1,200

Degree of Deformation and Specimen Size

These two factors clearly involve similar problems and are considered together.

Progressive deformation of a metal raises its strength and hardness, and normally reduces the temperature at which recrystallisation starts, provided that some true cold working is being applied. Owing to the hardness, strength and low elongation of tungsten, and to the narrow range between ultimate tensile strength and elastic limit, working at low temperatures offers serious difficulties, except for extremely light reductions, so that swaging and drawing are always carried out at above red heat. It was thus difficult to estimate the true degree of cold deformation to which wires received after commercial working had been subjected. However, although absolute degrees of deformation could not be estimated with any precision, experiments were made on wires after differing amounts of drawing in the laboratory.

Fig. 1 shows curves of tensile strength against annealing temperature for Sample *A*, tested at 300, 200 and 100 μ diameter. It is seen that the shape and position of the curves have been considerable modified by the various degrees of working.

The temperature of onset of secondary recrystallisation was raised from *ca.* 1,400° to *ca.* 1,700° C. as the wire was reduced from 300 to 100 μ diameter, and the step-like shape of the curve—most marked for the 300 μ diameter wire—became more gradual for the small wire sizes. These characteristics were reflected in the microstructures (see Figs. 2–6). Also, whilst in wire at 300 μ diameter the formation of small equiaxed grains readily occurred, coarsening of 100 μ wire had been by direct transition from fine to coarser elongated grains.

Fig. 7 illustrates the effect of deformation on a sample having a high secondary recrystallisation temperature. This behaved in the same way as the previous sample, although to a much smaller degree, the finest wire again having the highest secondary recrystallisation temperature. The changes in microstructure for the three samples of wire were very similar to each other, and also to those for Sample *A* at 100 μ, with a direct transition from the columnar structure to a coarsened state. (Figs. 8, 9).

Unfortunately, the brittle nature of fully annealed tungsten precludes it as a starting material for the examination of the effects of small deformations at low temperatures. Nevertheless, it was considered of interest to examine the recrystallisation behaviour after applying small cold deformations to wires annealed at as high a temperature as possible, the limit being set by the coarsening temperature of the material used. The deformation applied was by extension. Elongations for tungsten are very low at room temperatures, but were found to rise to a maximum near 200° C. This behaviour is in accord with some other observations on the elongation—temperature relationship, but completely in disagreement with the observations of Millner,[11] who finds a monotonic fall in elongation with rise in temperature over this range. Elongations at 200° C. were measured on wires after annealing at various temperatures, and Fig. 10 shows the plots of elongation at room temperature and at 200° C. for Sample *D*. It is seen that there is a very sharp maximum in the elongation at 200° C. with annealing temperatures between 1,850 and 1,900° C. (which is just below the coarsening temperature), with an extension at this temperature of 21%.

Since softening, as measured by tensile strength, of tungsten wires does not occur on annealing at temperatures below 600° C. for times as long as 10 minutes, the elongation tests at 200° C. taking place in about 2

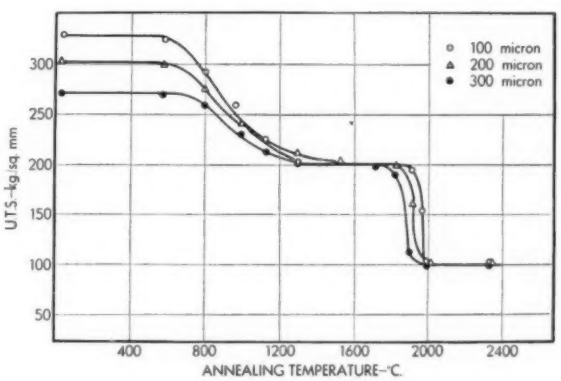

Fig. 7.—Effect on the tensile strength of Sample B, of annealing for 10 minutes at various temperatures.

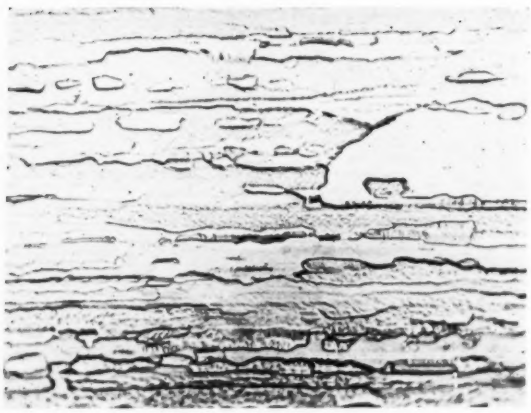

Fig. 8.—Microstructure of 300 micron wire of Sample B, annealed for 10 minutes at 1,800° C. × 1,200

Fig. 9.—Microstructure of 300 micron wire of Sample B, annealed for 10 minutes at 1,850° C. × 1,200

minutes can reasonably be considered as true cold working. The deformation was found to be distributed evenly over the length of the specimen. In this way, therefore, cold extensions of 10% could be applied to wires after annealing at 1,800° C., and specimens so treated were tested by plotting the U.T.S./annealing temperature curve. It was expected that "enforced secondary recrystallisation"[12] would develop, but, in fact, the coarsening behaviour was hardly influenced other than by lowering the temperature of its initiation by *ca.* 50° C. It may be significant that small deformations in a direction differing from that during the drawing (e.g. bending) has a much more marked effect.

When wire is to be drawn to finer sizes, it is often the practice to precede further working by a high temperature treatment within the primary recrystallisation range. Samples were therefore annealed at a diameter of 180 μ, under conditions of time and temperature just not severe enough to cause coarsening, and thus provided specimens with low degrees of deformation after virtually complete primary recrystallisation. Fig. 11 gives curves for a series of wires after annealing at 180 μ and then drawing to various diameters.

Neglecting the effects on primary recrystallisation, the secondary recrystallisation has been altered in perhaps a rather unexpected manner. It has been described earlier how the coarsening temperature tends to rise with increase in deformation, but it is here seen that the secondary recrystallisation temperature has fallen (at least within this range) progressively with increased working.

It was felt necessary at this stage, to resolve whether differences in recrystallisation behaviour between coarse and fine wire were due to effects due to deformation *per se*, or to the geometry of the specimens.

To obtain further information, Sample *B* was taken at 480 μ diameter and electrolytically etched and polished to give samples at 320 μ and 160 μ diameter, then drawn to 200 and 100 μ diameter, respectively; a further sample was drawn normally to 300 μ diameter. In this way, three drawn wires were produced of different sizes, having a similarly worked surface, but with equal degree of deformation.

These wires were tested in the usual manner, and although scatter was somewhat greater than normally found, a single curve could be drawn through all the points, indicating that wire size alone is not an important factor.

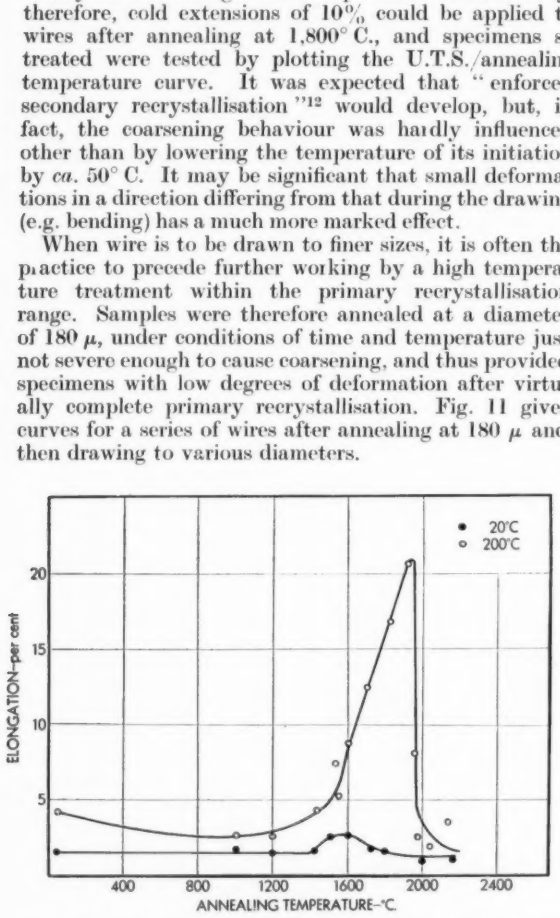

Fig. 10.—Effect on the elongation of 200 micron wire of Sample D on annealing at various temperatures.

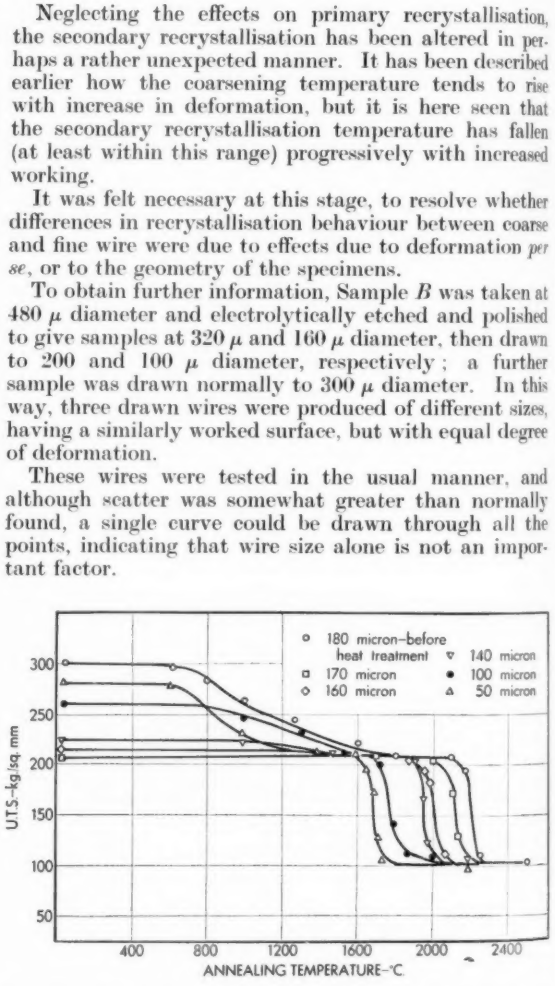

Fig. 11.—Effect on the tensile strength of Sample L of deformation after annealing at various temperatures.

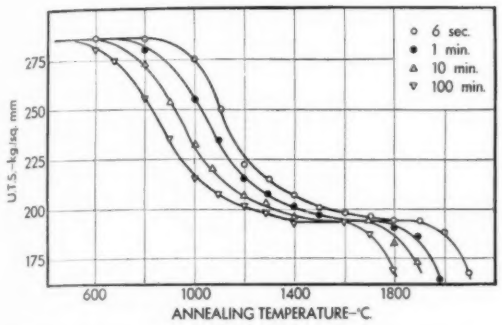

Fig. 12.—Effect on the tensile strength of Sample D of the time of annealing at various temperatures for primary recrystallisation.

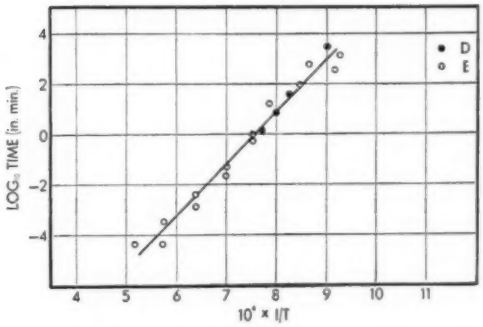

Fig. 13.—The relation between time and temperature for a given degree of recrystallisation (U.T.S. 240 kg./sq. mm.) in Samples D and E.

These results thus show that the stability of the columnar structure is progressively enhanced by continued working, and this is reflected in a raising of the coarsening temperature and a greater tendency to coarsen without intervening equiaxing. But, when a high temperature annealing treatment is interposed during the working, the coarsening temperature is lowered as working proceeds.

Interstage Annealing

Interstage annealing was carried out on wires at 215 μ diameter at temperatures of 800, 1,000 and 1,300° C.; it was found that the effects were confined to the processes of primary recrystallisation, having no influence on coarsening.

Time of Heating

Fig. 12 shows a family of curves drawn through values of tensile strength plotted against annealing temperature for four heating times, for the first stage of recrystallisation. These and further results were re-plotted to give curves of tensile strength against log. time for different temperatures. From these latter curves, estimates were obtained of approximate times for one-half primary recrystallisation, i.e. for tensile strength of 240 kg./sq. mm., and these times were re-plotted. (Fig. 13).

From this plot an estimation of activation energy for the combined factors controlling primary recrystallisation, recovery, etc., was estimated at *ca.* 100 k. cal./g. atom. This value is an "apparent" activation energy: the subject is considered in greater detail subsequently.

The same procedure was repeated for the higher temperature range covering secondary recrystallisation (see Fig. 14). Owing to the much steeper slope of the curve compared with that for primary recrystallisation, scatter was considerable, and the precision was hardly sufficient to justify the estimation of the activation energy for this process.

A study of the microstructures of the wires subjected to these tests for the effects of time and temperature revealed that short times at high temperatures tended to favour direct coarsening from the columnar structure, while longer times at low temperature permitted some equiaxing.

These results for Sample *D* illustrate the behaviour of a material with a coarsening temperature which is neither particularly high nor particularly low. Sample *L*, tested at a diameter of 180 μ, was taken as an example of a batch with a very high, sharply defined coalescence temperature, and the effect of the time of heating was investigated, giving the results listed in Table I. It is evident that, in this case, the time of heating had little effect on the coalescence temperature; a possible explanation is offered below.

TABLE I.—ULTIMATE TENSILE STRENGTH OF SAMPLE *L* AFTER HEATING

Temperature (° C.)	Heating Period					
	6 sec.	20 sec.	1 min.	3 min.	10 min.	32 min.
2,000 ..	—	—	200	200	200	180
2,100 ..	200	200	200	200	200	135
2,200 ..	125	120	115	120	115	130
2,300 ..	115	115	110	110	110	—

Amount and Nature of Additives

An obviously important variable, indeed perhaps the chief factor, is the nature of the starting material, and this especially applies to the nature and amount of additives.

Samples *B*, *D* and *L* referred to earlier were all suitably "doped" with small amounts of additives to produce "non-sag" wire; Sample *A* was a "non-sag" wire from an external source. In Table II are listed analyses made on the samples using the spectrographic method[13] for most elements.

It was observed that there was poor correlation for the most part between starting materials and impurity content in the final wires. This has been observed

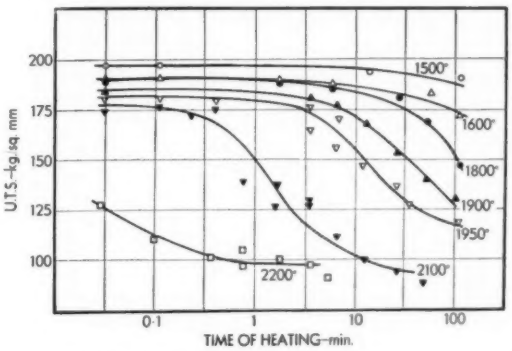

Fig. 14.—Effect on the tensile strength of Sample D of the time of annealing at various temperatures for secondary recrystallisation.

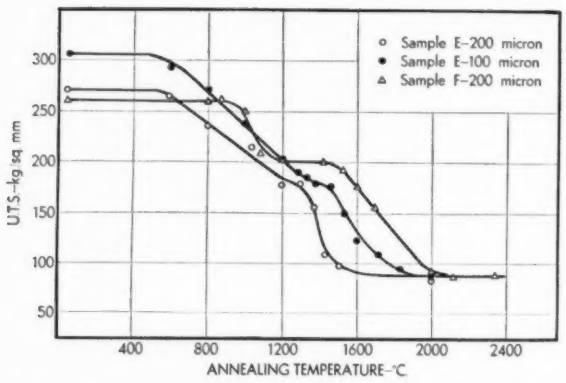

Fig. 15.—Effect on the tensile strength of undoped (E) and thoriated (F) wire of annealing at various temperatures.

elsewhere, together with the fact that accidental contents of certain elements may exceed that due to deliberate addition, and yet the latter process imparts valuable properties. Millner[11] gives an ingenious and well supported explanation for this finding, referring to differing manners of distribution of the impurities.

To examine the influence of impurities, batches with different additives were investigated. Sample *E* was a specimen of tungsten wire made from metal free from deliberate additions (see Fig. 15). It may be seen that a marked step associated with onset of secondary recrystallisation is almost, but not entirely absent. Also that, as in other samples, further deformation had raised the temperature of the step. Fig. 16, for 200 μ diameter wire shows that coalescence was preceded by marked equiaxing; grain growth of the equiaxed units was relatively slow with rising temperature.

A sample of thoriated wire, Sample *F*, containing 0·70% thoria by analysis was tested and the resulting curve is shown in Fig. 15. It is seen that, compared with other materials, a considerably higher temperature was required to initiate primary recrystallisation, but that this was completed over a small temperature range. The second fall in tensile strength, due to secondary recrystallisation, was gradual.

TABLE II.—ANALYTICAL DETAILS OF SAMPLES

Sample	Mo %	Fe %	SiO_2 %	Al_2O_3 %	Mg %	Ni %	Others %
A	0·01	0·005	0·016	0·004	—	—	—
B	0·02	0·005	0·01	0·006	0·001	0·002	—
D*	0·05	0·013	0·16	0·006	—	0·001	—
E	0·02	0·004	0·02	0·002	0·001	0·001	—
F	0·07	0·003	0·004	0·002	0·001	0·002	0·70 ThO_2†
G	0·06	0·002	0·005	0·002	0·001	0·002	—
H	0·06	0·003	0·013	0·003	0·002	0·003	—
I	0·06	0·003	0·005	0·18	0·001	0·003	—
K	0·06	0·005	0·016	0·003	0·001	0·002	{0·022 Zr {0·017 Zr‡
L*	0·04	0·002	0·08	0·010	—	0·001	—
N	0·02	0·004	0·013	0·003	0·001	0·001	—
O	0·02	0·004	0·006	0·004	0·001	0·001	—
P	0·02	0·004	0·005	0·003	0·001	0·001	Th ‡
Q	0·02	0·004	0·005	0·003	0·001	0·001	0·026 Zr
R	0·02	0·003	0·004	0·003	0·001	0·001	Ca ‡

* — Analysed on powder—remainder as wire.
† — Estimated chemically.
‡ — Found but not estimated.
Zr was detected in those cases where thorium was present.

The photomicrographs showed large quantities of an insoluble phase, distributed into "stringers"; even after annealing at 2,000° C. there was no sign of agglomeration of the inclusions or modification from their original disposition. At 1,500 and 1,600° C., coalescence of the tungsten grains had started but the grain size was small compared with other doped specimens, and even at 2,100° C. growth was slight (Fig. 17).

To throw further light on the effect of additives, five batches of wire were fabricated from the same silicated metal powder, all being similarly treated throughout the processing. Sample *G*, a material with 0·025% alumina added to the metal powder acted as a standard. Other batches were made from the same starting material (*G*) but with extra additions as follows:—

Sample *H* .. As Sample *G* with 0·025% magnesia
Sample *I* .. As Sample *G* with 0·5% alumina
Sample *K* .. As Sample *G* with 0·025% zirconium hydride

Results of the annealing tests for these batches are given in Fig. 18.

The photomicrographs of Sample *I* were found to be similar to those for the thoriated wires described above; they showed large quantities of inclusions, and the formation of a structure after recrystallisation of small equiaxed grains, which were subject to gradual growth with rising annealing temperature. From this it appears that an addition of 0·025% of alumina was more effective in retarding coalescence than twenty times this amount.

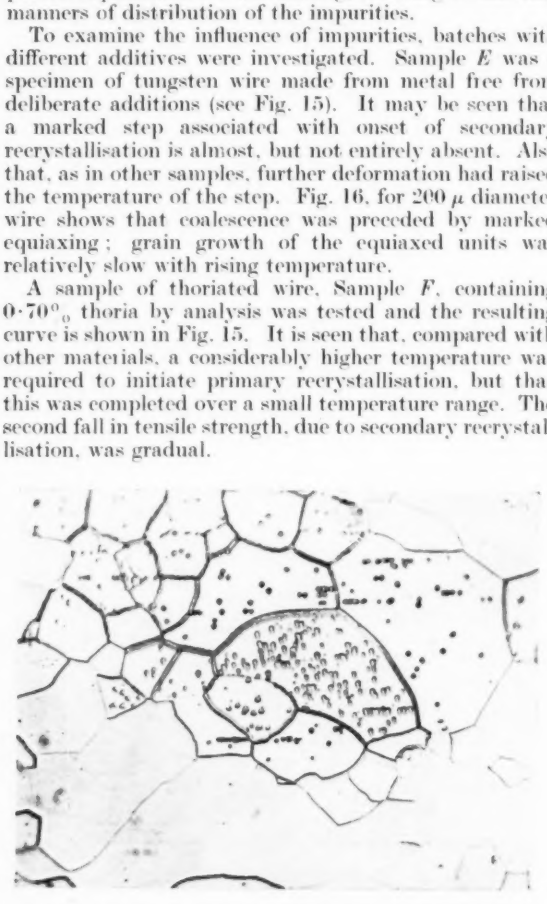

Fig. 16.—Microstructure of 200 micron wire of Sample E, annealed for 10 minutes at 2,000° C. × 1,200

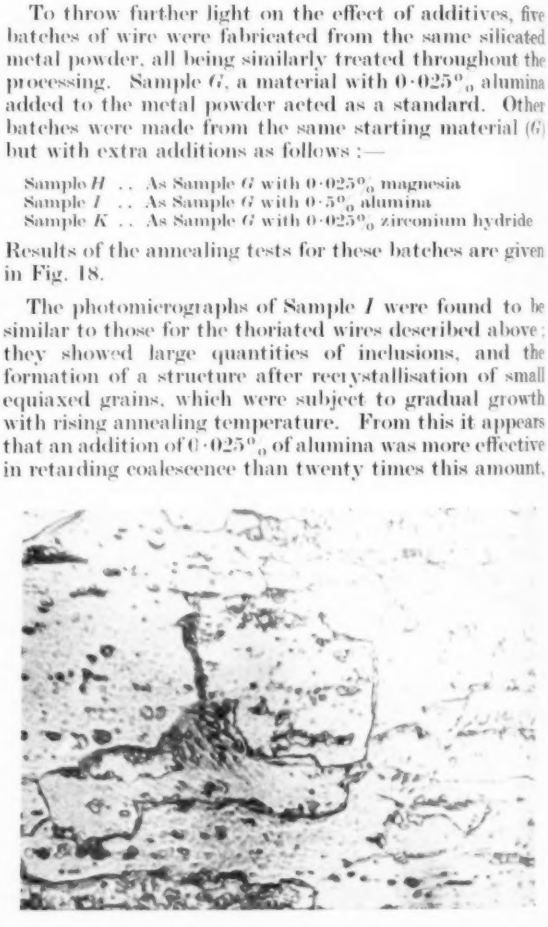

Fig. 17.—Microstructure of 200 micron wire of Sample F, annealed for 10 minutes at 2,100° C. × 600

TABLE III.—COARSENING TEMPERATURES FOR 10 MINUTE HEATING PERIODS

Sample	Temperature for U.T.S. of 180 kg. sq. mm. (° C.)	Temperature for U.T.S. of 115 kg. sq. mm. (° C.)
N	1,975	2,025
O	2,000	2,100
P	2,000	2,250
Q	1,950	2,000
R	1,700	2,100

and this disparity is more marked when the final contents are compared.

Sample *K*, containing an addition of zirconium was characterised by resistance to recrystallisation both in the primary and in the coarsening ranges. In Sample *K*, the zirconium was added to the metal powder as zirconium hydride but analysis gave :—

Total Zr .. .. .. 0·017%
Metallic Zr .. .. 0·011%

Soluble impurities, and especially the refractory metals molybdenum and tantalum,[14] tend to raise the softening temperature of tungsten ; any zirconium in solution may be expected to behave similarly ; this would explain the high primary recrystallisation temperature.

A further series of samples was produced by making additions in equimolecular ratios to a silicated metal powder from a similar source to that of Sample *G*.

Sample *N* with 0·025% alumina
Sample *O* with 0·010% magnesia
Sample *P* with 0·065% thoria
Sample *Q* with 0·030% zirconia
Sample *R* with 0·014% lime

The usual tests were made on the wires after drawing to 180 μ diameter, but to avoid confusion from plotting the results on a single graph, the coarsening temperatures for heating times of 10 minutes are set out in Table III. There are several points of interest in these results :—

(1) While the coarsening temperature of each batch is high, there is some tendency for the temperature to be highest for the most refractory oxide additions, bearing in mind interaction with residual silica.

(2) Sample *P*, with 0·065% thoria, behaved quite differently from Sample *F*, with 0·7%, having a much higher coarsening temperature.

(3) The addition of zirconium in Batch *Q* in such a manner as to form oxide rather than metal (as in Sample *K*), produced a wire which behaved quite normally. It should be noted particularly that the microstructures of Sample *Q* showed none of the signs of equiaxing seen in Sample *K*.

Fig. 19 shows the results of inhomogeneous distribution of dope material : although nominally of the same composition as *K*, the manner of recrystallisation for *K*1 is seen to vary according to the local concentration of zirconium.

Conclusions drawn from the effect of additives were that :—

(*a*) The coarsening temperature is raised by the addition of insoluble impurities, and the additives are then more effective as they are more stable and refractory.

(*b*) Zirconium appears to have a substantial influence on recrystallisation processes, rendering them more sluggish. Present only as oxide, it acts solely as an inhibitor for coarsening.

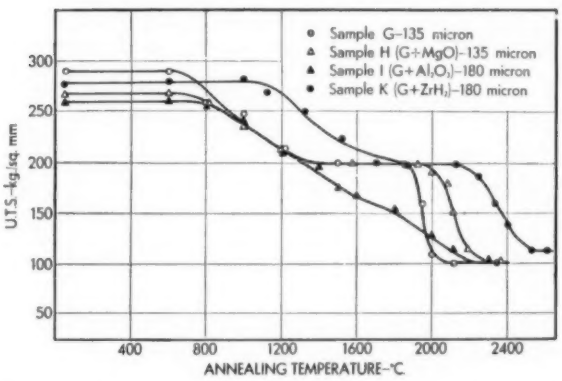

Fig. 18.—Effect on tensile strength of annealing variously doped materials.

(*c*) Material free from additives (Sample *E*) has a low resistance to initiation of coarsening, but nucleation is so high as to lead to moderate grain growth.

(*d*) Material with an excess of additives has a low resistance to initiation of secondary recrystallisation, but mechanical obstructions restrict final grain size.

Surface Condition

Inasmuch as recrystallisation commenced predominantly within the interior of the wires, little effect could be expected from the removal of surface layers. This was confirmed, but after removal of some 10–20 μ from the surface by electrolytic polishing, the mechanical properties were improved with increases in tensile strength up to 30% over untreated wires. The effect appeared to be associated with removal of surface discontinuities leading to notch brittleness.

X-Ray Results

Examination of several specimens was made by X-ray diffraction. In all cases, whatever the material or its condition (drawn, recrystallised, or with advanced secondary recrystallisation), there was a high degree of preferred orientation with the [110] direction parallel to the wire axis. (See refs. 6, 7, 15, but also 16).

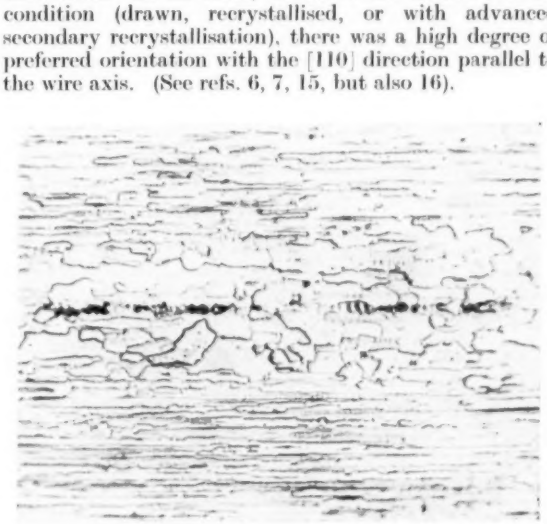

Fig. 19.—Microstructure of 180 micron wire of Sample K1 showing inhomogeneous distribution of additive. × 1,200

Cold Rolled Tungsten Sheet

A sample of tungsten sheet 30 μ thick was recrystallised and found to behave in an unexceptional manner; small, roughly equiaxed, grains formed during primary recrystallisation and these grains grew slowly in the normal way. The sheet was made from undoped metal and had a degree of preferred orientation somewhat lower than in wires; the [110] axis lay in the rolling direction, and the grains were orientated with a statistical maximum of grains having (100) planes in the rolling surface, but with some rotational scatter about the [110] direction.[17]

A second sample having a much more perfect texture, after annealing above 1,500° C. for 10 minutes, shattered on bending, with fractures running at 45° to the rolling direction. But despite the change in properties, X-ray diffraction studies on this sample after treatment up to 2,000° C. failed to reveal any recrystallisation, except in small regions at the surface.

(To be continued)

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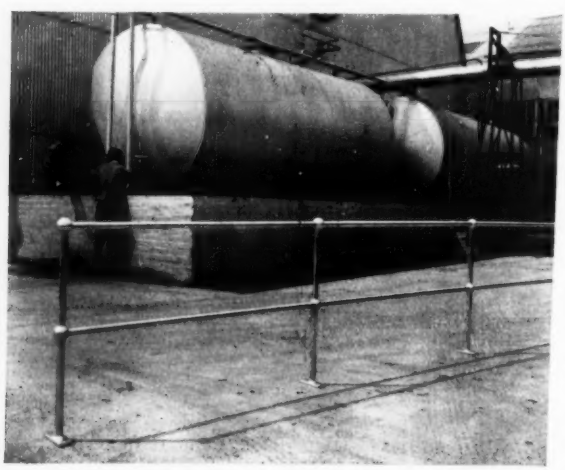
Aluminium Handrails

HANDRAILING is erected for convenience and for reasons of safety; it is an essential that is taken for granted. On first consideration, the material of which handrailing is made, including the stanchions that support it, may seem unimportant, provided it is strong enough and of reasonable price. Cast or forged steel standards and tubular steel railings, galvanised or repeatedly painted to forestall the effects of weathering, have been in use for many years. Less extensively, aluminium has also been used, such installations dating back thirty years or more.

Now the development of a forged aluminium stanchion, which is used with extruded aluminium tubing, merits careful consideration on account of the advantages it offers. The most important of these is durability; an alloy has been chosen that combines the necessary strength with excellent resistance to atmospheric corrosion whether in industrial or sea-coast environments. For this reason, and since aluminium's natural appearance is pleasantly appropriate (aluminium paint is often used on steel handrailing), the metal can be left unpainted. In fact, once the aluminium handrailing has been installed, it requires virtually no maintenance whatever. Lightness, aluminium's best known characteristic, though not so important here, adds considerably to the ease of installation, which is further enhanced by the facility with which the aluminium tubing can be formed on site.



The stanchions, which are produced by Northern Aluminium Co., Ltd., who also extrude the tubing to be used with them, will be available in three sizes, one of which is already in production. Standing 3 ft. above ground level, it is of the two-ball type (the lower ball being centrally placed between the upper one and the ground) accommodating railings of $1\frac{3}{4}$ in. maximum outside diameter. The stanchion tapers in diameter from $1\frac{1}{2}$ in. at the foot to $1\frac{1}{4}$ in. at the base of the upper ball, and it weighs about $6\frac{3}{4}$ lb. At present, two styles of foot are available and the range will be extended as required. In addition to the natural finish, the stanchions are also offered polished and anodised, in which form they are an economically attractive alternative to plated steel for installations where appearance is all-important. The alloy used is Noral B51S, in the fully-heat-treated or solution-heat-treated condition (conforming to B.S. 1472 : HF30), and the tubing is normally supplied in the same alloy in the fully heat-treated condition.

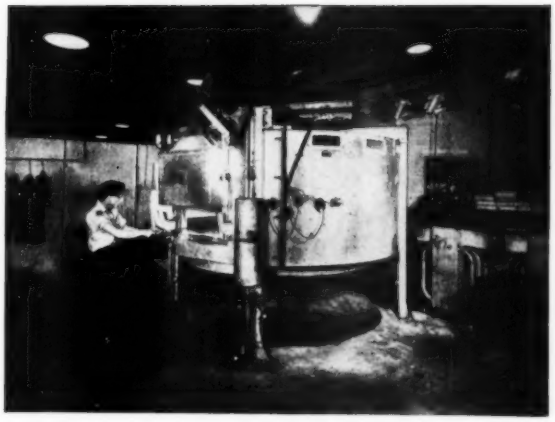
Of a pleasing appearance well suited to any environment, it would seem that aluminium handrailing of this sort will find its greatest justification in chemical works and nuclear power plants, in shipbuilding and the erection of promenades, and in sewage disposal plants, where the fullest advantage can be gained from its high resistance to aggressive atmospheres.

Uddeholm's New Division

IN order to extend their service to users of hard metal in the production engineering field, Uddeholm, Ltd. (the British associates of Uddeholms AB, the large and old-established Swedish mine-owning and steelmaking concern) have inaugurated a new division. Known as the Tool Division of the company, it will be concerned with the sale and development of small tools as used in production engineering, with particular emphasis on the application of Uddia brand hard metal in such components as tips, tools, dies, rolls, tube mandrels, sand blast nozzles and wear-resisting parts. The headquarters of the division will be at Crown Works, Northwood Street, Birmingham, 3. (Tel. ; CENtral 8971, Telex : 33/151).

Heat Treatment in Restricted Spaces

Advantages of Continuous Furnaces



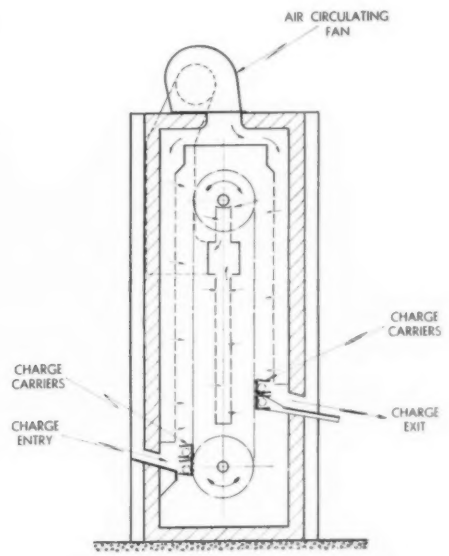
G.W.B. rotary hearth furnace for gear hardening.

PRESENT day building costs and the current increase in the rateable value of industrial premises make it imperative that the best use should be made of factory floor space. This problem is one of many which confront the production engineer, the most efficient disposition of plant and machinery representing a study on its own. On considering modern heat-treatment plant, it is evident that there is a growing trend towards continuous as against batch type equipment, frequently installed in the production line. It is often the case that a continuous furnace will occupy more space than a batch type unit and, therefore, much thought has been devoted to producing furnace designs which aim primarily at reducing the required floor space to a minimum. Some interesting examples of space saving continuous heat-treatment equipment are given in the following paragraphs dealing with recent electric furnace designs.

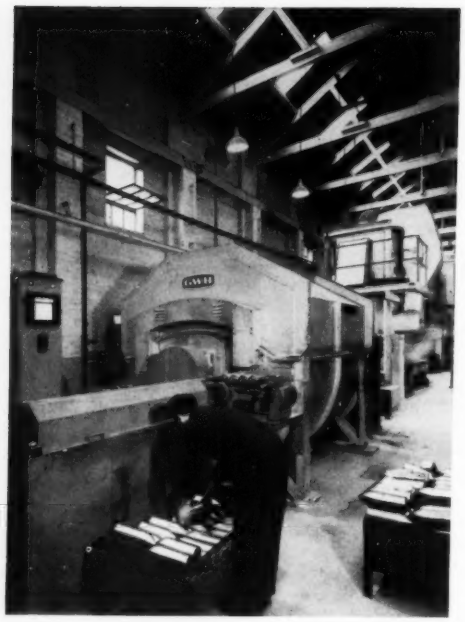
G.W.B. vertical conveyor furnace with cooling chamber wall removed to show the conveyor.

Rotary Hearth Type

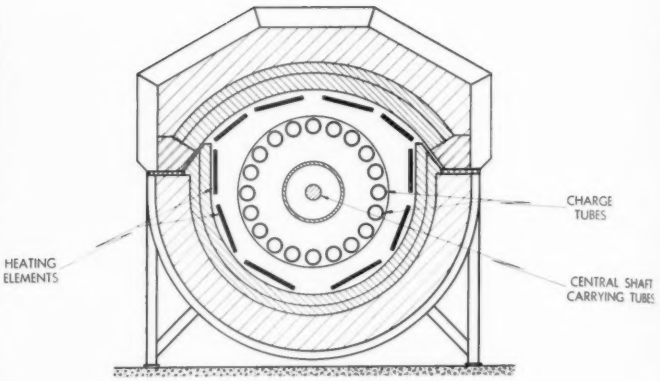
One of the most common furnace designs which is extremely compact and economical from the space saving point of view is the rotary hearth furnace. It will be apparent to engineers with only the slightest knowledge of furnaces that if the equivalent hearth heating length of a rotary unit were in the form of a " straight-through " furnace, a considerably greater floor space would be required. An added advantage of the rotary hearth furnace is that all the moving portion of the hearth is continuously within the heating chamber so that, in the absence of repeated heating and cooling of conveyors, the only thermal losses are those from the opening of the



Section through vertical conveyor furnace.



The charging end of a G.W.B. rotating tube pusher feed furnace.



Section through rotating tube pusher feed furnace.

loading and unloading doors, and the small degree of radiation loss through the insulating brickwork. It is also a comparatively simple matter for minor repairs to be carried out in the event of damage to the hearth by the simple means of lowering from the fixed furnace body. Another point in its favour is the fact that the loading and unloading doors are adjacent, and these two operations can be carried out quite easily by one person.

Vertical Conveyor Type

Another furnace design which takes up very little floor space is the vertical conveyor type. This can be likened to a horizontal pan conveyor unit set up on its end. As will be seen from the accompanying illustration, the charge pieces are loaded into a small opening towards the bottom of the furnace, rolling into swivel mounted baskets slung between chains running over driven sprockets. The conveyor speed can be varied, and at the discharge point a tilting device is usually fitted to tip the charge into trays or on to a further conveyor for subsequent processing. An air circulation system can be incorporated in the heating chamber in the case of the lower temperature units, and should controlled cooling be required as part of the heat-treatment, specially constructed cooling sections, either lightly insulated or water jacketed, with forced cooling air, if necessary, can be fitted in the half section of the furnace where the downward movement of the components takes place. While the rotary hearth furnace mentioned earlier can be used for processing relatively heavy items, the weight of the charge is a limiting factor in the case of vertical conveyor units. In one typical case, the base measurements of an existing vertical conveyor furnace are 5 ft. 6 in. × 6 ft. 3 in., and as the height of the equipment is over 21 ft., one side of the furnace being used for heating and the return side for cooling, this installation would have been over 40 ft. long if it had been of the horizontal type. Although due attention must be paid to the weight factor, it will be of interest to note that one vertical conveyor furnace is successfully tempering steel projectiles $3\frac{3}{8}$ in. diameter × $15\frac{1}{2}$ in. long and weighing 30 lb. each.

Rotating Tube Type

A further interesting example of space-saving heat-treatment equipment is the rotating tubular feed pusher furnace, which, while of novel design, has been well proved in actual practice. Briefly, the furnace body itself consists of a roughly cylindrical refractory lined casing, in which rotate a series of tubes fixed to a central shaft. The rotation of the tube-carrying shaft is effected by means of a paul ratchet arrangement, so that the tubes are positively located, each in turn, against the loading hole in front of the pusher ram. As each charge piece, usually a round billet or cylindrical projectile, is pushed into the awaiting tube, so the component at the far end of the tube is discharged : the shaft then rotates to bring the next tube up in line with the loading hole, and the operation is repeated. In a typical unit, 20 heat-resisting steel tubes are arranged around a cast heat-resisting steel shaft body at a pitch circle diameter of 36 in. The movements of the rotating tubes and the pusher gear are, of course, carefully synchronised to prevent mechanical damage to the installation. Apart from the very great saving in floor space, this type of unit is capable of an extremely high degree of thermal uniformity, since the heating elements can be arranged all round the internal refractory lining.

The continuous strip annealing furnace, in which the strip travels up and down in a vertical plane within a tower like furnace, is another example of a continuous heat-treatment unit taking up little floor space in relation to the output obtained. To obtain high outputs of annealed strip of a relatively narrow width would require horizontal furnaces of unwieldy length, particularly where a lengthy controlled cooling cycle is required, resulting sometimes in a cooling chamber 2–3 times the length of the heating chamber. A limiting factor however, which must be remembered when considering the tower type of furnace, is the available height within the factory.

All the foregoing installations are fully continuous in operation, but it should not be forgotten that the well

known semi-continuous bell furnace is also extremely economical in its floor space requirements, in relation to the output that can be obtained. In addition, flexibility of operation may be obtained, enabling varying process cycles to be carried out, and the complete absence of any complicated mechanical gear is a recommendation to be borne in mind.

It will be seen from the examples quoted, that the modern furnace designer is not lagging behind in offering to industry compact equipment capable of large outputs at high efficiency. Production engineers are going to look upon the continuous furnace more and more as a machine tool which can be placed in the production line alongside other metal forming and processing machines.

Oxygen Enrichment in Iron and Steel Making

THE development of the use of oxygen in steelmaking processes has been going on for a number of years, and several methods are well established, but the use of oxygen in ironmaking is yet in its infancy. Several theoretical approaches have been made and appraisals published on the possibility of introducing reducing gases together with oxygen into iron-making blast furnaces but, so far, little work has been done and no results published. The introduction of an oxygen enriched blast into the conventional blast furnace has, however, been the subject of experimental work in several countries, and has opened up the possibilities of improved output at economical costs.

About 90% of British steel is made in open-hearth furnaces. Although new steelmaking processes have recently been developed, the capital cost of replacement of the open-hearth method would be high, and manufacturers in this country, are therefore, constantly seeking to carry out modifications to open-hearth furnaces so that productivity can be increased and costs decreased without the need for replacement of plant. A practical method of achieving these aims is to introduce oxygen, and a review of experiments which have been carried out in Britain and in other countries in the use of oxygen in both iron and steelmaking was given recently by Mr. J. L. Harrison, Technical Advisor on Iron and Steel Processes, British Oxygen Gases, Ltd., in an address to the Newcastle Branch of the Society of Instrument Technology.*

Blast Furnace Trials

Describing trials in blast furnaces which took place at the Weirton works of the National Steel Corporation of America, Mr. Harrison said that they were conducted over a long period with additions of oxygen ranging up to 4%. During the experiments, the following results were obtained. There was an increase in the production rate, proportional to the percentage of oxygen in the blast, provided the total blast volume was constant. There was also an increase in the calorific value of the top gas to 107 B.T.U./cu. ft., which rendered it more valuable for general steelworks purposes. No damage to refractories resulted from enrichment: three out of four furnaces at Weirton were used and each completed a campaign to produce 3½ million tons of iron. The most satisfactory experiments were those in which enrichment was coupled with control of the moisture in the blast. The amount of moisture required for each percentage enrichment appeared to be approximately 3½ grains/cu. ft. The effect of the moisture was, presumably, to limit hot spot formation in the tuyere zone, and to produce a small quantity of hydrogen in the stack which would be conducive to reduction. The present Weirton practice is to share the available oxygen between furnaces, giving each an enrichment of 1·8%. Even in a range of up to 4%, this suggests that the most effective use of oxygen is at low percentage enrichment. Oxygen enrichment is now established practice at Weirton.

The American results can be compared with Russian experiments at the Novo Tulsk Metallurgical Combine. The practice there is based upon a predominantly sinter burden, the latter containing about 50% iron, and the furnace output is about 1,800 tons of basic iron per day. Experiments were carried out with oxygen enrichment of up to 24%. An increase in working rate was observed, amounting to 6·7% with 23·3% oxygen in the blast (2·5% above normal). This was less than would have been anticipated on the basis of the results obtained at Weirton.

Furnace behaviour was described as steady, although there was some disturbance due to poor quality sinter. It was claimed that smooth operation could be obtained without adding moisture to the blast. While this may be true, it is thought that the relatively large increase in productivity in the American trials may be due to the reducing action of hydrogen in the furnace stack. Reports of the Russian experiments stated that there was no significant change in iron quality or in furnace lining. A complete cost analysis was carried out and it was decided that, with oxygen at tonnage cost, blast enrichment would be an immediate practical proposition.

The higher hearth temperatures and the reduced gas velocity which result from enrichment suggest that the practice may be particularly suitable for the manufacture of ferro-alloys and in low shaft furnaces. For the reduction of the oxides of manganese, chromium and silicon with carbon, higher temperatures are required than are necessary in the case of iron. This suggests that the use of blast enrichment may be of particular value as far as coke economy is concerned. The high hearth temperature will give better recovery of the metal being reduced, since the reaction is allowed to proceed further. Reduction in dust losses and increase in top gas temperatures will also be experienced.

Trials at the Duquesne works of the United States Steel Corporation involved enrichments of up to 27% oxygen. An increase in production of 29% was experienced, together with a reduction of 9% in the coke rate and 8% in dust loss. In common with Weirton, it was necessary to increase the moisture content of the blast. Using two large furnaces, output has been increased from 650 tons per day to 850 tons.

Open Hearth Practice

For some years it has been realised that oxygen is exceedingly valuable for the decarburisation of low

(*Continued on page* 192)

* "Certain Aspects of the Use of Metallurgical Oxygen" by D. W. Pitts, M.Sc. and J. L. Harrison, F.I.M. (British Oxygen Gases Limited).

Some Properties of Zinc-Aluminium Alloys

By K. P. Scott, B.Sc. (Eng.), A.R.S.M.

Imperial Smelting Corporation, Ltd.

In this article the author presents the results of mechanical tests on zinc-aluminium alloys containing 2–50% aluminium. In the cast state, optimum mechanical properties are obtained with a 4% aluminium alloy. Alloys containing more than 10% aluminium have poor impact strength, and shrinkage porosity is present in alloys containing more than 20% aluminium

IT is well known that the common zinc die casting alloys in use today are based upon the zinc-aluminium system, having been developed by The New Jersey Zinc Co. in America and selected from many thousand compositions exhaustively investigated over a period of thirty years as best fulfilling the requirements for alloys suitable for use in die casting[1]. These alloys conform to the requirements of composition for Alloys A and B of B.S.1004 : 1955 (Zinc Alloys for Die Casting) and contain approximately 4% aluminium and 0·05% magnesium. In the case of Alloy B, 1% copper is also present. Both alloys are made from high purity zinc of composition not less than 99·99% purity, the impurities in which must meet the requirements of B.S.1003 : 1955 (High Purity Zinc).

Whilst a considerable amount of work has been undertaken and published concerning the properties of the zinc-aluminium system, there does not appear to be available any information on the effect of increasing aluminium content on the mechanical properties of zinc. The object of the work to be described, therefore, has been to investigate the properties of zinc-aluminium alloys containing aluminium in amounts varying from 2 to 50%. Reference to this investigation has already been made in a paper presented at the Paris International Pressure Die Casting Conference held in May, 1957.[2]

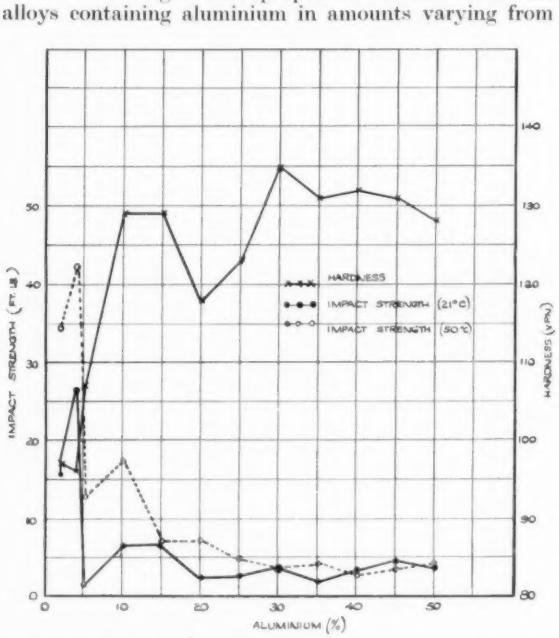


Fig. 1.—Results of mechanical tests on zinc-aluminium alloys containing increasing amounts of aluminium.

TABLE 1.—IMPACT AND HARDNESS TEST RESULTS ON ZINC-ALUMINIUM ALLOYS.

Alloy (% Al.)	Impact Strength (ft. lb)		Room Temperature Hardness (V.P.N.)
	21°C.	50°C.	
2	15·6	34·5	97
4	26·4	42·2	96
5	1·6	13·0	107
10	6·7	17·7	129
15	7·0	7·2	129
20	2·5	7·2	118
25	2·5	5·0	125
30	3·9	3·9	135
35	2·0	4·2	131
40	3·3	2·8	132
45	4·4	3·5	131
50	3·9	4·2	128

Preparation and Casting of Alloys

All alloys were prepared from high purity zinc (99·99+%) and super purity aluminium. An addition of 0·04% magnesium was made to each melt. The alloys were gravity chill cast in the form of bars ¼ in. square and 6 in. long, the casting temperatures employed being 30–40°C. above the liquidus temperature in each case.

It was found that those alloys containing 2–5% aluminium possessed good casting properties and resulted in good quality bars being obtained. The 10% and 15% aluminium alloys were inferior to these, and many of the bars cast had to be rejected owing to surface defects. Above 20% aluminium the castings improved, but were not as good as those obtained from the 2–5% aluminium alloys. The fluidity of the alloys containing more than 5% aluminium (the eutectic composition) appeared lower than that of the alloys with less than 5% aluminium. After casting, the bars were allowed to age at room temperature for one day before testing.

Mechanical Testing

For test purposes the cast bars were cut into 3 in. lengths. Impact strength was then determined on a Charpy machine of 52 ft.lb capacity, tests being made both at 21° and 50°C. It is well known that pressure die castings in Alloy A of B.S.1004 undergo a reversible change in impact strength at about 0°C., and since this change occurs at slightly higher temperatures for gravity castings, the selection of the higher testing temperature was considered to be desirable in order to eliminate any chance effects arising from this transition. A total of ten specimens of each melt was tested at each temperature. Hardness values were obtained on a broken impact specimen using a Vickers Hardness Tester with a load of 10 kg.

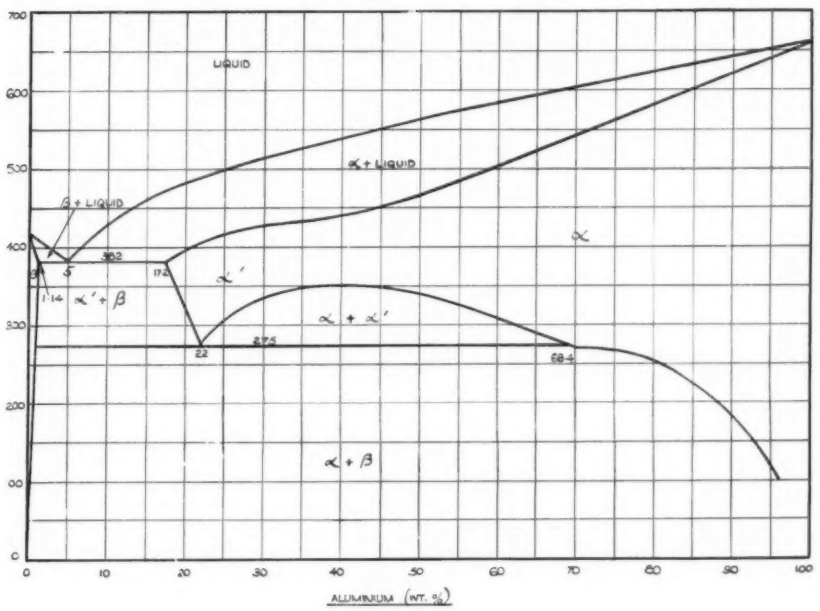


Fig. 2.—Equilibrium diagram for zinc-aluminium alloys. The temperature scale is in °C.

Results

The results obtained are shown in Table I. The impact strength reported is the average of the middle two values obtained after arranging the ten results in ascending order of magnitude.

From Fig. 1 it is apparent that there is a rise in hardness when the aluminium content of the alloys increases from 4 to 10%. Within the same composition range, the impact strength attains a maximum value at 4% aluminium, decreases rapidly at 5% and recovers slightly with the addition of 10% aluminium. Reference to Fig. 2 shows that the 5% aluminium alloy corresponds to the eutectic composition of the zinc-aluminium system.

Additions of aluminium beyond 10% produce no further increase in hardness, which, in fact, ultimately decreases at the 20% aluminium content. This point would appear to be coincident with the eutectoid transformation in the system (22% aluminium, balance zinc : 275°C.) Further amounts of aluminium again result in increasing hardness, reaching a maximum at 30% and thereafter declining gradually to the upper limit of composition examined. Beyond 10%, additions of aluminium cause a steady decline in impact strength.

It is of interest to record that the castings made from alloys containing more than 20% of aluminium all showed porosity cavities when examined metallographically. This porosity appeared to be greatest at about 35% aluminium ; Fig. 3 shows an unetched photomicrograph of a casting of this composition.

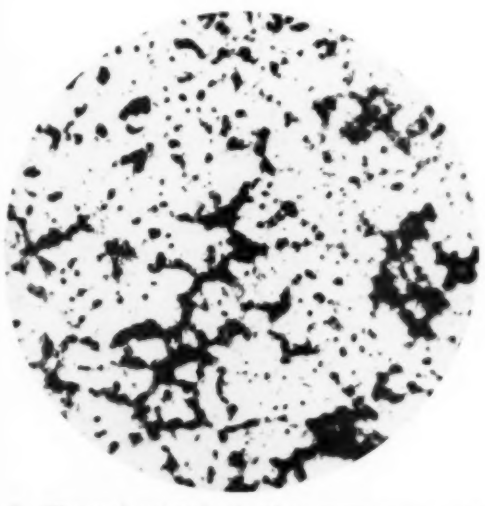


Fig. 3.—Photomicrograph showing porosity in a zinc-35% aluminium casting. ×300

Conclusions

From the work described, it is apparent that in the cast state, optimum mechanical properties are obtained with a composition of 4% aluminium, balance zinc. This alloy corresponds to the standard zinc die casting alloy in use today. Alloys containing 10–15% aluminium whilst being harder, lack the impact strength possessed by the 4% aluminium alloy. Even higher amounts of aluminium, up to 50%, result in alloys possessing moderate hardness, but having very poor impact resistance, and which are subject to shrinkage porosity when cast.

Acknowledgment

The author wishes to make acknowledgment to Mr. M. Joyner formerly of University College, Cardiff, and at present at Bristol University, for carrying out the experimental work described, and to the Directors of the Imperial Smelting Corporation, Ltd., for permission to publish this work.

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2 "The Influence of Aluminium in the Zinc Die Casting Alloys." L. A. J. Lodder and K. P. Scott. Paper presented at the 2nd International Die Casting Conference, Paris. May 20-24, 1957.

Low Ductility in Cast Carbon Steel

By T. A. Cosh,* B.Sc., A.R.C.S.T., F.I.M.
and W. J. Jackson,† M.Sc., A.R.I.C., A.I.M.

Investigation of a number of individual cases of low ductility in plain carbon steel castings suggests the presence of certain types of inclusion as the cause. The authors discuss the ways in which the particular inclusion types may be avoided, whether they arise as a result of steel-making procedure, or mechanical or chemical attack upon refractories.

VERY occasionally some steel foundries have experienced difficulty in attaining a high degree of ductility in plain carbon steel castings, and this paper is based upon investigations by the B.S.C.R.A. of several individual cases of this nature. In the cases investigated the failures were attributable to two causes : (*a*) indigenous inclusions, and (*b*) exogenous inclusions. Indigenous inclusions are those involving metallic compounds of oxygen, sulphur or nitrogen which separate out of the molten steel. They occur mainly as deoxidation products. Exogenous inclusions may arise from slag entrapment, from occlusion of refractory material, or from the interaction of constituents of the molten steel with refractory materials.

Indigenous Inclusions

A large amount of literature exists on the subject of deoxidation, and yet improper deoxidation arising from a failure to appreciate and apply basic principles is still occasionally encountered. Steel is deoxidized only when the total oxygen content is present in an innocuous state. It is not sufficient to consider a deoxidant in terms of its affinity for oxygen relative to the oxygen affinity of iron. That is, of course, an important consideration. More important, however, is the nature of the deoxidation products formed. If these products remain present in the steel, then it is imperative that they should not adversely affect the properties. It is better, therefore, to aim at deoxidation products which will migrate from the steel to the slag. Elimination of the deoxidation products from the steel will depend on the characteristics of the products in respect of solubility, fluidity, density, shape and size. Consideration must also be given to the affinity of the deoxidants for other elements, especially sulphur and nitrogen, and in particular the effect of oxygen content on such affinities. Otherwise the effect of the deoxidant might be to combine to form and precipitate sulphides or nitrides rather than oxides.

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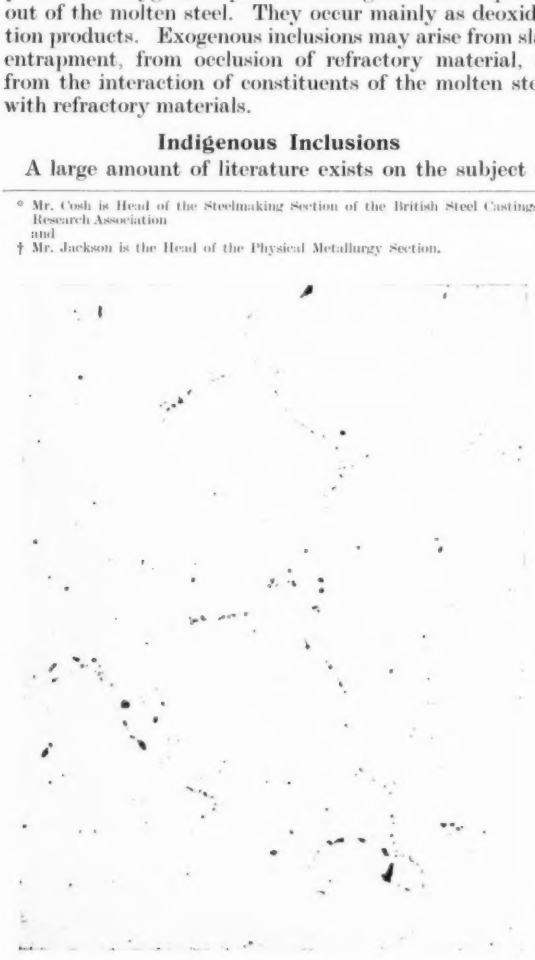

Fig. 1.—Duplex grain boundary sulphides (Type II) with some alumina. Unetched. × 150

Deoxidation Power and Inclusion Type

Deoxidants can be classified in the range of weak to strong according to oxygen affinity. The strength of the deoxidant is also related to the type of inclusion formed, the inclusion type classification being that enunciated by Sims and Dahle.[1] The weaker deoxidants tend to form Type I inclusions and the stronger deoxidants Type III, viz :

Inclusion	*Deoxidant*
Type I	Silicon, manganese, vanadium
Type II	Aluminium, titanium, zirconium, boron, magnesium
Type III	Aluminium, zirconium, magnesium

This classification is applicable when each of the deoxidants is used individually. Both the deoxidation power and the inclusion type are modified when deoxidants are used in combination.

Deoxidation with the weaker deoxidants normally results in Type I inclusions, which are globular and randomly dispersed thoughout the steel. This type of inclusion gives very good mechanical properties, but unfortunately the use of a weak deoxidant will not safeguard against the formation of pinhole porosity. To prevent pinhole porosity adequately, strong deoxidants must be used. Furthermore, they must be used in quantities great enough to ensure the formation of Type III inclusions which are angular but randomly dispersed. Their presence therefore permits the attainment of mechanical properties approaching those obtained when only Type I inclusions are present. Type II inclusions, which are elongated and occur predominantly at grain boundaries, arise from the improper use of strong deoxi-

dants, and generally result in failure to meet mechanical test requirements.

It is clear, therefore, that in practice the most desirable form of inclusion is that of Type III. In order to obtain Type III, strong deoxidants must be used, which can also give rise to Type II inclusions. The latter, however, are formed only when the deoxidant is present in insufficient quantity. By this it is meant that, although the weighed-out addition may be quite adequate to produce Type III inclusions, the technique of making the addition might involve wastage and insufficient recovery of the deoxidant. This is a major problem with aluminium, which readily floats and wastes itself on the surface of the molten steel. Thus, Type III inclusions may not be assured. However, the use of a calcium-silico-manganese addition with the aluminium addition will eliminate any tendency to Type II formation, and good mechanical properties will be obtained.

The deleterious Type II grain boundary inclusions (Fig. 1) occur with a low residual aluminium content, in the range 0·01% to 0·03%. The formation of Type III inclusions is assured when the residual aluminium is in excess of 0·04%. Therefore, allowing for some wastage, the normal aluminium addition should be of the order of 0·10%, i.e. $2\frac{1}{4}$ lb/ton.

When more than one deoxidant is used, the nature of the deoxidation products is influenced by the order of the addition of the individual deoxidants. For example, the solubility of manganese sulphide, MnS, is greatly influenced by the FeO content of the steel, and is at a maximum when the FeO is at a minimum. Thus, the addition of manganese to an already fully killed steel can result in the solution of an excessive amount of MnS, which will be precipitated later on solidification (Fig. 2). It is therefore preferable to add ferro-manganese prior to any addition of ferro-silicon and essential to make any ferro-manganese addition, before an aluminium addition.

Fully killed steel in which some of the sulphur is present as aluminium sulphide will gradually re-oxidize on standing or holding in the ladle. The Al_2S_3 will be oxidized to Al_2O_3, as will any residual metallic aluminium, and this will promote conditions favourable to the formation of Type II inclusions.

Deoxidants must therefore be added in increasing order of oxygen affinity, with the final addition as near to casting as possible. The use of calcium-silico-manganese will assist in the dispersion of clustered inclusions, and is particularly recommended when the time between tapping and casting is likely to be prolonged. The addition of calcium-silico-manganese should be in the same proportions as the aluminium addition. The recommended quantities of deoxidants, subject to specification requirements, and the order of their addition is :

Deoxidant	*Amount Added (lb./ton)*	*Method of Addition*
Ferro-manganese (78%)	20	To bath
Ferro-silicon (75%)	12	To bath
Aluminium	2–$2\frac{1}{2}$	To ladle
Calcium-silico-manganese	2–$2\frac{1}{2}$	To ladle

Exogenous Inclusions

This type of inclusion can arise from three causes :

(*i*) slag entrapment ;

(*ii*) occlusion of refractory particles arising from mechanical or chemical attack ;

(*iii*) products of reaction between molten steel and refractories.

Fig. 2.—Globular MnS or Fe-MnS eutectic with lamellar sulphide (Type II). Unetched. × 500

The nature of the inclusions will depend therefore on the steel-making process—acid or basic ; and open hearth, electric arc, high frequency or converter—which will determine the types of refractory used and the slag make up.

Slag Entrapment

Inclusions arising from this cause always have the same composition as the finishing slag. They occur as the result of poor steel-making practice in respect of temperature of operation, slag viscosity and failure to stir or rabble. Any one or any combination of these factors will result in slag inclusions. Even when adequate steps have been taken by means of temperature control, slag composition and good furnace operation to eliminate inclusions from the metal as tapped, the use of lip pouring ladles with an inadequate slag dam will allow the slag to enter the mould and nullify the earlier precautions.

Occlusion of Refractory Materials

Into this category fall the inclusions arising from the erosion of the furnace lining, furnace launders, ladle linings, ladle nozzle assemblies and sand from runners and moulds. Such erosion may be mechanical, due to the flow of metal over the refractories, or chemical, due to the interaction of slag and refractory.

Erosion of furnace linings arises from the use of inadequate refractories, excessive operating temperatures, or corrosive or overfluxed slags. Erosion of furnace launders and ladle linings may be due to the use of poor refractories, inadequate drying, the use of materials with a very low dry strength, bad patching, or impingement of excessively hot metal. Some erosion of the nozzles of stoppered ladles is unavoidable, but it can be minimized

Fig. 3.—Exogenous inclusions with entrapped MnS at top left-hand corner. Unetched × 120

by the use of adequate refractory material and good stopper control. The erosion of sand in the mould is dependent on the quality and strength of the sand mix, and the design of the mould and its gating system. All of these causes can be minimized by good practice in each of the departments concerned.

Interaction Products of Steel and Refractory

Such reactions occur mainly when the steel is in the killed condition. In such circumstances the metallic constituents of the steel will tend to react with oxides of the refractory and produce duplex oxides as inclusions, often by the mutual fluxing of two oxides. For example, manganese in killed steel will tend to reduce silica in siliceous refractories. The MnO so formed has a severe fluxing action on refractories of firebrick quality resulting in $MnO \cdot Al_2O_3 \cdot SiO_2$ complexes (Fig. 3). Such a tendency can be minimized by the use of good quality ladle linings, and also by reducing the manganese content in relation to the silicon content of the finished steel, as far as the specification will allow.

Summary

Low ductility in cast carbon steel can arise from a variety of causes, each of which can be minimized by good technical control. The precautions necessary to assure freedom from inclusions involve more than one department in the steel foundry and can only be guaranteed with stringent supervision and inter-departmental co-operation.

Acknowledgment

The authors are grateful to the Council and the Director of the British Steel Castings Research Association for permission to publish this paper.

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1 Sims, C. E., and Dahle, F. B., *Trans. A. F. A.*, 1938, **46**, 65-103.

Oxygen Enrichment in Iron and Steel Making

(*Continued from page* 187)

carbon steel made in open-hearth furnaces, and the chemistry of the injection of oxygen for this purpose is now fairly well understood. The Steel Company of Wales have found it possible to take a sample for carbon and sulphur, add lime in accordance with the latter figure, and blow with a predetermined amount of oxygen based on the carbon analysis. The steel is ready for tapping on completion of the blow. Heat is generated by metalloid oxidation and this, together with the physical effect of the oxygen jet, fluxes the lime and provides the high basicity and temperature needed for sulphur removal. Oxygen consumption by the Steel Company of Wales at Margam is approximately 150 cu. ft./ton of steel. This is the figure for blowing from about 0·40% carbon at a rate ranging up to 1,000 cu. ft./min. of oxygen (i.e. 5 cu. ft./ton/min.). This type of practice is now well established. Although the action is a little vigorous initially, it calms as the carbon falls.

At the Zaphorozhstal Works in Russia, there are ten 185-ton open-hearth tilting furnaces using 70% hot metal. There, decarburisation is the normal practice, jetting through the roof being the standard technique used. If suitable jets are used, no difficulty is experienced in refining steel down from quite high initial carbons.

The East Chicago works of the Inland Steel Corporation have attempted the oxygen conversion of hot metal containing up to 1% phosphorus. Initial experiments were carried out in a small side-blown converter holding 800 lb. of metal, oxygen being lanced into the melt from the side. Final figures showed a production rate of 40·1 tons/hr., an oxygen consumption of 2,100 cu. ft./ton, and an ingot yield of 86%.

The coming of the European Common Market requires that British steelmaking costs should remain as they are at present—competitive with those elsewhere in Europe. If new processes are capable of producing steel at a cheaper rate than is possible with the open-hearth furnace, however, it may be necessary for existing plant to be replaced.

European Documentary Leaflets

With a view to meeting the need for accurate and easily digestible information on the background to the setting up of the European Free Trade area, some sixty leaflets are to be produced this year at quarterly intervals. They will cover such subjects as the European common market; the European Convention of Human Rights; Africa and Europe; and Atomic Energy in Europe; and will have a standard format; a special file will be provided with them for easy reference. They may be obtained from Young Europe, 57 Dean Street, London, W.1., at a cost of 15s. for the series.

Cold-Heading

Recent Swiss Developments in Progressive Headers

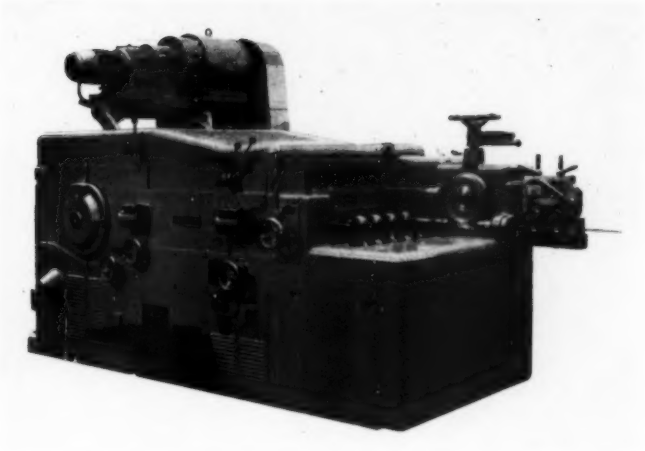


Fig. 1.—Hatebur BKA 3 progressive cold-heading machine.

THE manufacture of standard bolts, either by the cold-heading or other process, is a somewhat unremunerative line of business even in the best of times, and most manufacturers in this industry have to look to " specials " as the profitable side of their business. In the United States, it has been possible to cheapen production of both standard and special bolts by the use of a header unit which incorporates both a shank-pointing and thread-rolling machine, and such a combination has notable advantages as long as each unit runs on the same size of work continuously. The output, however, is such that there are few individual manufacturers outside the U.S. having a sufficient volume of business in individual sizes to maintain such a combination machine working for any prolonged period upon a single size ; and it is precisely to meet the lower scale of European production that the Swiss firm of F. B. Hatebur, of Basle, has designed the progressive header shown by the photograph reproduced in Fig. 1. This is a four-blow progressive header having one shearing and four subsequent heading stations, though minus pointing and thread-rolling attachments, but of the universal type on which, in addition to standard and special bolts, many other intricate components can be mass-produced from coiled wire.

Whilst this type of machine is manufactured in three sizes, having capacities from $\frac{3}{8}$ to 1 in. diameter wire, the machine illustrated is the BKA–3, which weighs approximately 29 tons, absorbs 35 h.p., and has a combined heading tonnage of 180, the maximum diameter of wire being $\frac{3}{4}$ in. and the maximum length cut-off 6 in. It is capable of a working output of 30,000 pieces per 8-hour shift. Fig. 2 shows the sequence of operations on this machine in the production of a special bolt using double extrusion and head trimming. When cold-heading from wire of diameter identical to that of the shank, there is no cold-forming on the shank portion, which results in a considerable difference in tensile strength and elongation as between the head and the shank, thus necessitating a final stress-relief annealing operation. In the case of the method shown by Fig. 2, however, starting from wire of a larger diameter and reducing the whole length of the shank, the tensile strength of the shank is increased, and there is less tension between this portion and the head, so that no heat-treating operation is necessary, the bolts coming from the machine only requiring thread rolling and packing before despatch. The method also has the

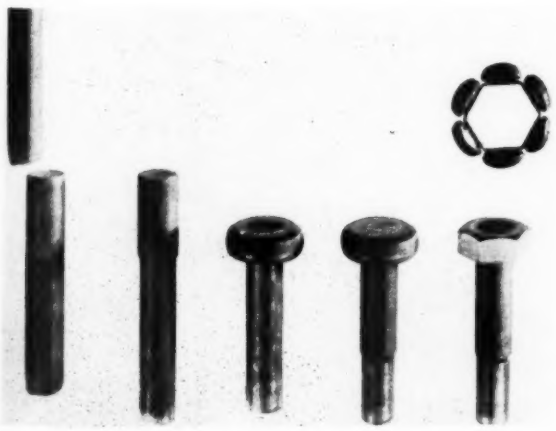


Fig. 2.—Sequence of operations in producing special bolt.

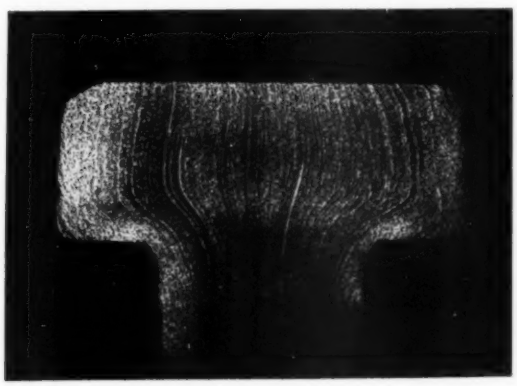


Fig. 3.—Fibre flow of bolt blank.

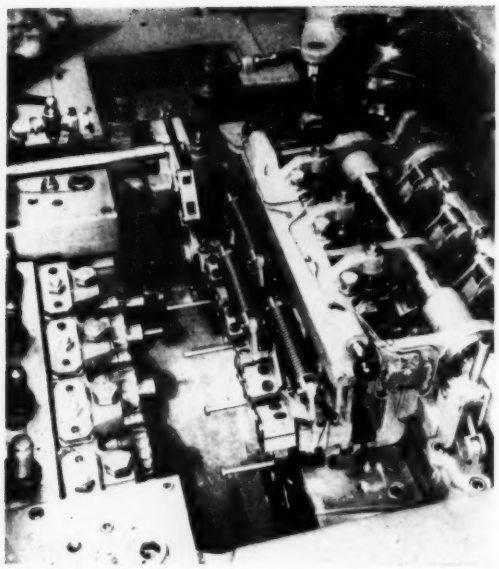


Fig. 4.—Tooling layout on Hatebur BKA 3.

advantage that various shank lengths can be produced by the same tooling, this being possible due to the fact that the shank reduction for thread rolling is made a single operation by the third blow. The process is stated to be especially suitable for the manufacture of high-tensile bolts, although in this particular instance, owing to the special tensile strength required for this type, a heat-treating operation would be necessary.

The advantage of trimming being combined in the one machine immediately following heading is an important one, as there is no chance of age-hardening of the heads as there was formerly, when pieces had to be stacked between the header and the separate trimming machine.

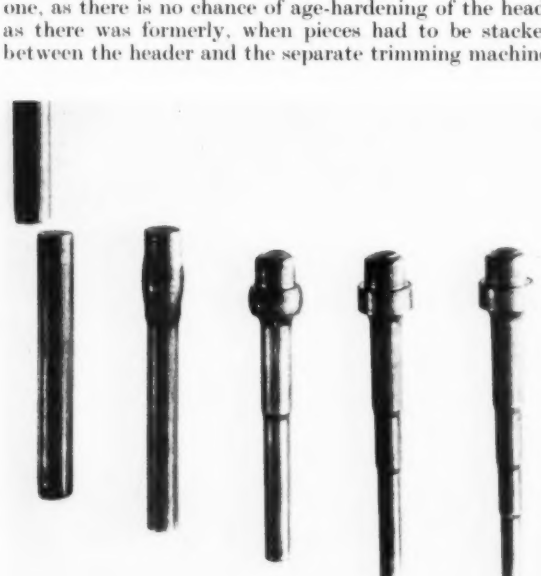


Fig. 5.—Sequence of operations in producing pedal spindle.

As the two processes are combined in the same machine, the production keeps in step. While there are some very fast double-blow machines in existence, it sometimes happens in bolt plants that the pace of production is set by the independent trimming machines, which cannot maintain step with the production of the latest type of headers.

Fig. 4 is a photograph of the tooling lay-out on a BEA-3 Hatebur progressive header. The opening and closing of each pair of transfer fingers can be timed to meet the individual requirements of each heading station. Each heading station has its own ejector lever and cam, thus allowing the use of specially developed cams when necessary. The last heading station can be set to any cold-forming operation or for trimming.

As already mentioned, the employment of this type of cold header is no longer confined to the manufacture of bolts, and the pedal spindle shown by Fig. 5 is typical of the many other classes of work which can be economically produced by this means. Output of the single Hatebur machine working on these pedal spindles is stated to be 80 pieces per minute.

New Sunvic Training School

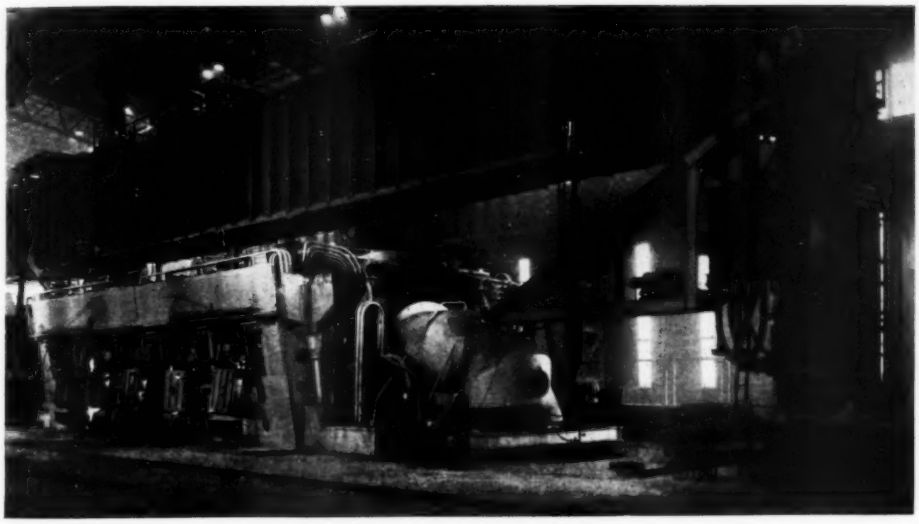
A TRAINING school to instruct customers' operators and maintenance staff in the use of Sunvic pneumatic and electronic control instruments has recently been opened by Sunvic Controls, Ltd., at their Harlow, Essex, factory. In duration and content the course is flexible, depending upon the degree of technical detail required by students, but it will not generally exceed one week. It will cover design, operation and applications of the pneumatic range of process control instruments which are widely used in the oil and chemical industries. In addition to lectures given by Sunvic technical staff, facilities are provided for practical instruction in the well-equipped laboratory which is a feature of the school. An interesting demonstration of Sunvic instruments under operating conditions can be given on a plant simulator and control panel. This has been specially devised for the school, and enables students to obtain first-hand knowledge of the operating efficiency of instruments. The school which has for some time been providing instruction in use of Sunvic multi-channel pulse height analysers will now be merged administratively with the new process control school, although courses will normally be quite separate.

Arc Furnace Exported

THE first of two 40 ton G.W.B.-Tagliaferri direct arc steel melting furnaces was recently shipped to Canada for delivery to the Canadian Steel Wheel Company, Montreal. The largest item for shipment comprised the actual furnace shell, which weighed over 50 tons. This presented quite a problem since, owing to its size, it could not be taken by road transport direct to the side of the Birmingham City which was moored at Cardiff docks. Accordingly, the furnace shell was taken to the only point accessible, and was then carried to the Birmingham City by means of a floating crane. The rest of the equipment was brought in the normal manner to the side of the ship for loading. This furnace represents the first half of a $500,000 order, the second furnace being due for shipment at the beginning of this month so that it can reach Montreal in time before the St. Lawrence freezes over.

Oxygen Increases Open Hearth Output

Full Scale Trials at Scunthorpe



General view of the Ajax tilting open hearth furnace at Appleby-Frodingham Steel Company showing oxygen lance entering the furnace at right through the steel casing which is designed to eliminate infiltration and thereby minimise the volume of waste gases to be cleaned.

IMPORTANT modifications to a 350-ton tilting open hearth furnace at the Scunthorpe works of Appleby-Frodingham Steel Company, a branch of The United Steel Companies Limited, have led to a substantial increase in steel output compared with normal furnaces, for approximately the same cost per ingot ton of steel. This has been achieved by the use of oxygen in the specially-adapted furnace.

Experiments in the oxygen lancing of open hearth furnaces have been carried out at Appleby-Frodingham since 1950 and have led to some exceptionally high rates of production. The higher outputs were partly offset, however, by such factors as severe roof wear and repeated blocking of the furnace chequers by dust. It became apparent that very considerable changes in furnace design and structure would be necessary in order to develop an efficient oxygen process, and that this could only be proved by full-scale trials. The demand for ingots to supply the company's rolling mills dictated a stringent time schedule for the reconstruction, and it was, in fact, accomplished in 27 days. Trials began on the modified furnace—which was given the code name of Ajax to distinguish it from conventional furnaces—in February of this year, and over 100,000 tons of steel have since been produced by the process. During a recent four-week period, this furnace produced a record 19,263 tons of steel, making over 5,000 tons per week in three of the four weeks. This is believed to be the highest output ever attained by an open hearth furnace in this country.

Details of Reconstruction

At the chimney end of the furnace, the first problem to consider was that of gas cleaning, in order to deal with the increased amount of suspended matter carried out by the waste gases while using oxygen, and to avoid excessive atmospheric pollution. On grounds of economy in capital cost and very low air infiltration expected, it was decided to instal a dust removal plant with a capacity much below that for an open hearth furnace of comparable production potential. Available evidence indicated the bag filter type as the one most suitable for this experimental furnace.

In order to minimise the volume of gas to be cleaned, the whole structure of ports, uptakes, slag pockets, regenerators and flues in the reconstructed portion of the furnace was completely encased in steel up to the junction of the main stack flue in the valve arch. Flexible packing joints are provided at every junction and at every inspection or cleaning hole.

The Ajax furnace regenerators are much smaller than those on a conventional furnace, so that they can be blown easily and quickly with compressed air lances at frequent intervals. The height of the chequer filling is similar, but the maximum gas flow rate more nearly resembles that of a blast furnace stove than an open hearth regenerator. The small cross-sectional area of the regenerator has an additional advantage when using oxygenated air to assist combustion, in that no added oxygen is lost through the structural brickwork.

The area of the offtake from the furnace hearth was also considerably reduced for four main reasons: (1) to lift the furnace end bank as high as possible in order to minimise slag overflow when sponging; (2) to minimise the overall weight of the offtake and uptakes so that they can be removed by crane and quickly changed; (3) to minimise the cost of relining with all-basic refractory

bricks ; and (4) because the gas volume leaving the hearth during most of the operating time is much below that of the normal open hearth furnace.

Reducing the area of the offtake enabled two separate slag pocket and regenerator systems to be installed at each end of the furnace in the same space normally required by one set of systems. This means that all repairs below the furnace stage can take place while melting is in progress, so that a general repair takes only the time needed to rebuild the furnace hearth itself. In the slag pockets, it means cooler working conditions for the men who can do their job during the normal working day instead of on shifts.

All-basic bricks are used in the lining of the ports, uptakes, slag pockets, and the upper half of the regenerator chambers and chequers of the Ajax furnace. Both the slag pockets and regenerator chambers are cylindrical in cross-section, with a view to simplicity of construction, strength of design, and minimum volume of basic brickwork. To conserve heat and protect the steel structure, insulation is used from the ports to the chimney flue, consisting of 1 in. Dextramite slabs on the steel shell, then 4 in. hard insulating firebrick, with 13½ in. basic end arch bricks as the working lining.

Machine-faced chills are employed to ensure a tight junction between the tilting hearth and the port, the port itself being on wheels with a water-seal connection to the uptake. Electrically-controlled compressed air cylinders ensure the best possible contact between the chills. The oxygen lance is introduced to the furnace through the port at an angle of 27°. The lance is fixed to a movable, motor-driven carriage and withdrawn at each reversal of gas and air ; it can be changed in 35 minutes.

Coke oven gas burners are inserted in each corner of the tilting portion of the hearth instead of through the port ends : this helps both to avoid any enlargement of the port and the addition of more trailing flexes. Oxygen can be used to assist combustion when required and is fed into the burners through two holes just below each gas inlet. For rapid melting, this placing is not very efficient, but it is quite adequate for holding the bath temperature at the end of refining, when burning-in dolomite, or heating-up scrap.

The thermal input of the furnace is low by design and, without oxygen, it would produce steel at probably no more than 10–12 tons per hour compared with 25–35 tons per hour when on oxygen.

Comprehensive controls and instrumentation are provided, all valves being centrally operated by electric pushbuttons to achieve the rapid manipulation which is essential to the successful operation of the process. The controls enable oxygen to be used in three ways : through the water-cooled lances, through the coke oven gas burners, or through the regenerators as an addition to the air. The latter is possible because of the freedom from infiltration.

Experience gained during several months of continuous operation of the Ajax furnace has proved the value of the process in raising steel output without incurring heavy capital expenditure and at no greater ingot cost.* But there are still many problems to be solved. Losses of iron in the slag, for example, are higher than in the normal Appleby-Frodingham practice. It still remains to be discovered how these losses can be reduced without slowing down the process : the furnace taps every 7 to 8 hours compared with normal practice of 14 to 16 hours and to retard it would only increase costs without a comparable saving in iron. An improvement in roof life is also sought, although the extra costs here are balanced by savings in " below stage " refractories. Finally, there is further work to be done on the life of the oxygen lance and the best gas velocity when lancing. The Ajax furnace is now the largest steel producing unit at Appleby-Frodingham and, in view of the rapid progress already made with its development, it seems unlikely that any of these problems will be found to be incapable of solution.

* Full details of the *Ajax* experiments are published in the September, 1958, issue *The Journal of the Iron and Steel Institute*, *cf.* " The Use of Oxygen in a Modified Tilting Furnace", by A. Jackson.

Cheaper Stainless Steel

PRICE reductions for a certain type of stainless steel used mainly in the motor car industry have been announced. A ferritic stainless steel strip, known as FI.17, in sizes up to but not including 15 in. wide, will carry reductions in price, ranging from £15 to £35 per ton, according to thickness and width. This is the first reduction in price of stainless steel since the war, and a spokesman of Firth-Vickers Stainless Steels, Ltd., Europe's biggest producer of these special steels, said today the cheaper prices are expected to stimulate the use of stainless steel in the motor industry. Stainless steel has been increasingly used over the past few years for hub caps, radiator grills, and window trim. Because of its anti-corrosive qualities and the fact that it is stainless throughout, stainless steel is finding favour for exterior car fittings which are subjected to weather conditions and which were previously made of chromium-plated materials.

New Laboratory Premises

(*Continued from facing page*)

department. Work study techniques are applied to all metal processing, handling and layout work. The establishment of time standards and bonus schemes for piece work operators and some job estimating is also carried out by this section.

Technical Service.—In addition to the above, the department provides a technical service to the sales, production and administrative departments. Members of the department are used as technical representatives for consultations with customers, and the technical service also includes answering queries, analysis and testing, and maintaining a small reference library. There are from eight to ten staff members in the department, the majority being specialists, but all are expected to do a wide range of investigational work.

Time study and quality control are housed in separate offices within the works. Much of the development work is done in the factory itself, using production machines, and this calls for the closest co-operation between development and production departments.

Each project is allocated to an individual engineer, whose responsibility it is to progress the work as rapidly as possible. The department's work is co-ordinated by the development manager, who is responsible to the managing director for the efficient running of the department. In addition to this, a committee chaired by the managing director and consisting of the heads of the Production, Engineering and Development Departments meets monthly to discuss progress on specific projects.

New Laboratory Premises for Aston Chain & Hook Co., Ltd.

An Integral Part of the Works

A view of part of one of the laboratories

THE opening of a new £15,000 laboratory block at the Erdington (Birmingham) works of the Aston Chain & Hook Co., Ltd., non-ferrous metal engineers, marks an important stage in the development of a policy which gives "the lab." a more than usually prominent role in the activities of the organisation. So far from being a somewhat narrow and specialist department, little concerned with matters outside research and development, the Aston company's new laboratory is now, formally and officially, the headquarters of a Development Department completely integrated with every form of works and staff activity from process control to work study. At the head of the department, as development manager, is Mr. Norman Harvey, formerly the company's chief metallurgist.

Mr. Noel Bond-Williams, the company's managing director, explained "This, even more than my own office, is the nerve centre of the factory, ready to tackle every kind of problem—and, as far as possible, before it becomes a shop floor headache. The old idea of a laboratory as a sort of ivory tower inhabited by men in white coats peering into microscopes and with little practical knowledge of management or shop floor conditions is, in my opinion, hopelessly out of date.

"Here we have a team of young scientists and technicians concerned always with the efficient running of the works and not just waiting to be given a lab-bench problem to sort out."

The company produces in the main extruded, rolled and drawn copper section bars, and in addition manufactures rolled brass, copper and bronze strip, wire, metal pressings and chain. With such a range of products it was considered essential to provide a competent technical control at all stages of production. This was the original function of the technical department and it began in 1938 with the establishment of a small analytical laboratory for control of the casting shop. During the following years the technological requirements of the industry have become more demanding, requiring greater intensification of effort on both technical control during manufacture and forward development of new and improved products and processes. This has led to the expansion of the department, which, as the Development Department, has now assumed responsibility for process control, inspection, work study, factory layout, and product and process development work.

The Work of the Development Department

The activities of the Development Department in the fields referred to are summarised below.

Process Control.—Routine chemical and mechanical testing of casting, drawing and strip mill products is undertaken, together with control of casting techniques and heat-weight calculation. A recent innovation, due to expansion of the strip-rolling facilities, is the appointment of an assistant responsible for all the technical aspects of rolled-strip production. This policy will be extended into other departments as occasions arise.

Quality Control.—Production foremen are responsible for the quality and inspection of the metal which they produce, but the standards of inspection to which they work and the quality control procedures adopted are laid down by the Development Department, which also acts as arbiter in doubtful cases. Inspection is done by statistical quality control and sampling techniques.

Product Development.—New shapes and new materials constitute the type of products which require experimental development work before being brought into production. In conjunction with this, drawing-die and press-tool design play a large part. This work is undertaken to discover the potentialities of new lines, to extend the range of existing lines, and to solve production problems on existing products.

Process Development.—This sphere of activity includes design, specification and commissioning of new plant and equipment, and the improvement of existing plant to increase productivity. Much of this type of work is done in conjunction with the engineering department. Plant layout is also an integral part of process development, and together with work study considerations is carried out to produce smoother work flow and better handling facilities.

Work Study.—Since much of the development work involves work study applications, there are obvious advantages in having this function within the same

(Continued on previous page)

NEWS AND ANNOUNCEMENTS

Sheffield Society Change

THE Sheffield Society of Engineers and Metallurgists was founded in 1894 under the Presidency of Sir Robert Hadfield, and for over sixty years it has provided occasions for the reading and discussion of papers closely related to the affairs of steelmaking in the Sheffield area.

At a recent review of the Society's activities, the Council had regard to the fact that, so far as purely metallurgical subjects were concerned, the metallurgists in Sheffield and the surrounding districts were well catered for by the Sheffield Metallurgical Association and the local section of the Institute of Metals, whilst general engineering was dealt with by the local branches of the Engineering Institutions. Attention was also drawn to the affiliation of the Society with the Iron and Steel Institute, a privilege which is also enjoyed by the Sheffield Metallurgical Association, and to the success which the Iron and Steel Engineers' Group of the Institute had achieved since its inauguration shortly after the war. It has therefore been decided that the Society will, in the future, devote the major part of its lecture programme to the same objectives, in co-operation with the Group. It is felt that in this way an important field of interest will be covered, and that there will be considerable support from engineers, designers and draughtsmen employed in local steelmaking and plant engineering firms.

In furtherance of this plan the Society has now published its programme for 1958–9, commencing with a paper on October 20th, on " Atmospheric Pollution in the Iron and Steel Industry." Other papers relate to rod and bar rolling, automatic press forging, a universal slabbing mill and continuous casting of steel. The Secretary of the Society, is Dr. J. F. Wallace, of The University, St. George's Square, Sheffield, 1.

Liquid Fuels Conference

THE Summer Meeting and Conference on " Major Developments in Liquid Fuel Firing 1948–1959 " will be held in Torquay from Monday, May 11th, to Thursday, May 14th, 1959. This Conference, organised by the Institute of Fuel, is intended to bring up to date the report of the Birmingham Conference on " Modern Applications of Liquid Fuels," held jointly with the Institute of Petroleum in 1948. Further particulars may be obtained from the Secretary, The Institute of Fuel, 18 Devonshire Street, Portland Place, London, W.1.

Foundry Development Laboratory

ONE of the largest and newest industrial installations for the development of advanced methods of foundry technology in the United States has been put into full-scale operation by General Electric of America. This is the Applied Research and Development Laboratory of the Foundry Department, which is providing the vital link between pure research developing new materials and processes, and the actual foundry use of such materials and methods as casting and improved production techniques.

The laboratory has a 9 ft. diameter vacuum degassing chamber—the first in use for foundry technology in the United States—to improve methods of eliminating gaseous impurities in molten cast metals. Other projects underway include the development of improved methods of vacuum melting and processing of cast alloys to reduce impurities and improve cast properties; improved precision casting methods; and methods for reducing the quantity of metal needed for rises, in castings.

Coal Science Lecture

SIR CHARLES GOODEVE, F.R.S., has accepted the invitation of the Council of the British Coal Utilisation Research Association to deliver the Seventh Coal Science Lecture on the subject of " Carbon: The Key to Metallurgy." The lecture will be delivered in the Lecture Theatre of the Institution of Civil Engineers, Great George Street, London, S.W.1, at 5.30 p.m. on October 15th, 1958. Admission is by ticket.

Exports of Iron and Steel Scrap

THE Board of Trade, after consultation with the Ministry of Power, announce that they are now prepared to consider applications for licences to export to any destination further limited quantities of iron and steel scrap. Applications stating grade, f.o.b. price and details of the firm order for the material, including the method and terms of payment, should be made to the Export Licensing Branch, Board of Trade, 2/14, Bunhill Row, London, E.C.1. on Form (F).

Nuclear Fuel Cycles Symposium

THE Institute of Physics, one of the constituent bodies of the British Nuclear Energy Conference, announces that it is arranging a Symposium in London on " Nuclear Fuel Cycles " on 22nd and 23rd January, 1959. The papers which have been invited will cater mainly for persons working in the field, and will deal with the applied physics aspects of the fuel cycles which form the basis of the immediate nuclear power programme. Abstracts (but not pre-prints) of the papers to be read or presented at the Symposium will be available early in January. The Symposium will be published in the Journal of the British Nuclear Energy Conference.

Further particulars and application forms for tickets to attend the Symposium are obtainable from the Secretary of The Institute of Physics, 47, Belgrave Square, London, S.W.1.

Birlec Standard Furnace Division

IN order to meet the increasing demand for standard furnaces, Birlec, Ltd., have set up a separate Standard Furnace Division under the managership of Mr. J. Penfold, previously Manager of the Furnace Department of the Morgan Crucible Co., Ltd., a step which has already shown its worth in reduced costs and improved deliveries.

Birlec Standard Furnaces include horizontal, vertical, and liquid bath batch type units, with shaker hearth and

mesh belt furnaces for continuous work handling. Catering for hardening, tempering, annealing, nitriding, brazing, sintering, die-casting, and other processes, with appropriate atmosphere control equipment, these furnaces bring to manufacturing and engineering industries the accumulated benefits of years of experience, research and development.

Quartz Agents

FLEISCHMANN (LONDON), LTD., of Chancery House, Chancery Lane, W.C.2. are the sole authorised agents and distributors for the United Kingdom of all quartz products manufactured by Heraeus Quarzschmelze G.m.b.H., Hanau, Western Germany, with the exception of optical quartz.

Personal News

F. H. LLOYD and Co., Ltd., announce the appointment to the Board of MR. W. R. COOPER (Assistant Managing Director of Lloyds (Burton) Ltd.), MR. W. H. NICKLIN (Sales Manager), MR. F. CLYMER (Foundry Manager) and MR. W. L. BEASLEY (Production Manager). Mr. Clymer has also been appointed a Director of Lloyds (Burton) Ltd., along with MR. M. C. LLOYD, M.B.E. (Assistant Managing Director of F. H. Lloyd and Co., Ltd.).

MR. S. FIELD has been appointed Chairman of Venesta, Ltd., in succession to MR. H. RUTHERFORD, who relinquished the Chairmanship on medical advice and in recognition of fifty years' distinguished service was created the Company's first Honorary President.

MR. J. HIGGINBOTHAM, after service in the Accounts Department and on the staff of the Foundry Manager, has been appointed Deputy General Manager of the Engineering Department of Edgar Allen & Co., Ltd. Other appointments in the Department include, MR. J. D. STUDHOLME as Assistant General Manager, MR. A. BRUNTON as Commercial Manager, MR. A. WALLIS as Chief Draughtsman (Production), MR. J. D. LEE as Chief Draughtsman (Development), and MR. S. CARTER as Chief Estimator.

GEORGE KENT, LTD., announce the appointment of a new Director, MR. T. P. W. NORRIS, lately Chief Personnel Officer of the Vickers Group, previously Labour Manager of I.C.I. Billingham Division, and a former President of the Institute of Personnel Management. Mr. Norris has been appointed Deputy Chairman. MR. R. E. HANDFORD, Deputy Managing Director, after forty-five years in the Company's service—twenty-five of them as a Director—has relinquished his executive functions, but retains his seat on the Board; and MR. W. A. HARTOP, who was appointed to the Board in 1943, has been appointed Managing Director: COMMANDER P. W. KENT, formerly Chairman and Managing Director, remains as Chairman. MR. W. GUY ARDLEY—who joined the Company in 1893 and became a Director in 1914—and MR. L. H. KENT, who joined the Company in 1913, and the Board in 1915, have retired from the Board.

MR. S. H. GRIFFITHS, Head of John Thompson Limited Group Research Laboratories, has been appointed a director of John Thompson (Wolverhampton), Ltd.

MR. A. P. M. PURDON has been appointed Operations Study Manager in charge of Central Operational Research and Work Study for the British Oxygen Co., Ltd.

MR. H. MORLEY, Deputy General Works Manager of Samuel Fox & Co., Ltd., a subsidiary of The United Steel Cos., Ltd., has been appointed General Works Manager of the Company, in succession to MR. J. D. JOY, who has been appointed Director and General Works Manager of Appleby-Frodingham Steel Co. with a view to becoming Deputy General Manager on January 1st next. MR. C. M. SLOCOMBE, Director and Chief Engineer of Samuel Fox, has been appointed Director of Engineering and MR. J. HAMMOND, Deputy Chief Engineer, becomes Chief Engineer. MR. H. J. STRONG, has joined the Company to work with the Chief Engineer with a view to becoming Works Engineer on January 1st, 1959.

MR. W. H. GLASS, O.B.E., who has been a Director of Thermotank, Ltd., the air conditioning engineers (Helen Street, Glasgow) for the past 25 years, has retired from the Board of the Company.

MR. C. E. HOLMSTROM has retired from the position of Joint Managing Director of Firth-Vickers Stainless Steels, Ltd., after forty-six years' service with the Company and its predecessors, and has been appointed a non-executive Director of the Company. Following Mr. Holmstrom's retirement, MR. J. T. W. DEWAR, Joint Managing Director of the Company, has been appointed Managing Director.

MR. W. L. BAKER, Manager of the Metal Finishing Division of The Pyrene Co., Ltd., has retired after thirty years' service with the Company. He is succeeded by MR. H. A. HOLDEN, who has had many years' experience in the Division. MR. H. F. PARSHALL, who has been with the Company for some twenty years, continues as Director in charge of the Division.

SAMUEL FOX & CO., LTD., a subsidiary of The United Steel Cos., Ltd., announce that their Research Department will, in future, be known as the Metallurgical Department, which reflects more accurately the nature of the Department's duties. Furthermore, the responsibilities of the Works Metallurgist, MR. R. WILCOCK, are to be broadened. He will be responsible to the Chief Metallurgist, MR. R. D. POLLARD, for all aspects of the company's internal activities in the metallurgical field—process and research metallurgy, the chemical laboratory, and the test house. To enable Mr. Wilcock to carry out his wider function, he will delegate much of his present work to a process metallurgist. MR. R. WHITTAKER, who was formerly employed in the Department, is rejoining Samuel Fox to become Process Metallurgist.

MR. W. M. FRAMES, of Johannesburg, formerly Chairman of Rand Mines, Ltd., has been appointed Chairman of The International Nickel Company S.A. (Proprietary), Ltd., South African subsidiary of The International Nickel Company of Canada, Ltd. Mr. Frames continues as a Director of Rand Mines, Ltd., and other companies. MR. J. HENDERSON recently joined Hepworth & Grandage, Ltd., Bradford, as Foundry General Manager. Mr. Henderson was previously employed by the Daimler Co., Ltd.

DR. M. F. JORDAN, formerly of Aluminium Laboratories, Ltd., has joined Murex Welding Processes, Ltd., as leader of the Fundamental Research Section of the Research Department. In this capacity he succeeds DR. W. D. BIGGS, who has now left the Company to take up an appointment in the Engineering Department of Cambridge University. Mr. H. J. WELLARD has also joined the Fundamental Research Team.

RECENT DEVELOPMENTS

MATERIALS : PROCESSES : EQUIPMENT

Metallizing Shop Truck

A STURDY, completely portable metallizing truck, developed by Metallizing Equipment Co., Ltd., is designed to give maximum mobility and efficiency to a metallizing installation. It permits the movement of all normal metallizing equipment, accessories and supplies necessary to do a good job, whenever and wherever required, so that there is no wasted time searching for accessories " borrowed " for other purposes. A built-in wire rack accommodates several coils of the various metal spraying wires which are in constant demand, and keeps them clean, compact and ready for use. The metallizing wire in use is fed from a standard wire reel table, through a straightening device, and at a convenient height. Shelves are provided for spare tools and necessary equipment.

The rigid chassis is mounted on four heavy-duty, wide-tread, rubber-tyred wheels, which permit easy movement with exceptional manœuvrability. The cylinders of gas and oxygen are carried within the wheelbase, which makes the truck extremely stable. With the standard metallizing accessories permanently attached, all the essential metallizing equipment is right where it is most needed. In its operational condition, the dimensions of the metallizing truck are 2 ft. 6 in. wide × 4 ft. 6 in. long. The truck is made completely collapsible for ease of transport, in which case the overall height is reduced to 1 ft. 2 in. The instrument panel is supplied drilled ready to take all necessary accessories used in metal spraying.

Metallizing Equipment Co., Ltd., Chobham, Surrey.

Eddy Current Crack Detector and Sorter

A NEW eddy current instrument has recently been added to the group of testing systems available from Magnaflux Corporation, Chicago. This instrument, known as the Magnatest ED-500, is designed to locate and determine the relative seriousness of defects in conductive materials ; it sorts mixed lots of ferrous and non-ferrous metals for differences in hardness, alloy, and heat treatment ; and measure the thickness of certain conductive and non-conductive coatings on conductive bases.

The ED-500 consists of an indicating unit, inspection probe, and power line cord. The sensitive inspection probe is part of an anti-resonant bridge which directly provides the desired signal and simultaneously compensates for spurious signals. As the probe passes over a crack or metallurgical difference, an unbalance occurs which is indicated by a needle deflection of a meter located on the front panel of the instrument. An all-purpose probe, angle probe, and spark plug port probe are available as standard accessories, and special probes can be designed as needed, to reach surfaces not easily accessible or to facilitate use on unusual contours.

A feature of this unit is its " lift-off " adjustment for stability of operation in testing semi-rough surfaces. With " lift-off " compensation, intermediate layers of dirt, oxide, pits, etc., up to a thickness of 0·020 in. can be tolerated before the accuracy of the meter reading is affected. The ED-500 is designed for use on a 60 cycle, 110 volt D.C. supply.

Magnaflux Corporation, 7300 West Lawrence Avenue, Chicago 31, Illinois, U.S.A.

Aerosol Fire Extinguisher

THE development of the Gwish Aerosol fire extinguisher makes it possible to locate an inexpensive fire fighting appliance beside each work-bench, machine or office desk, to deal with minor outbreaks of fire instantly before they become major conflagrations. Gwish, which is claimed to be less than one third the cost of normal type extinguishers, contains twelve fluid ounces of extinguishing liquid, released as a powerful jet spray by depressing the discharge nozzle ; release of the nozzle completely cuts off the jet. The spray douses flames instantly and carries for 6-ft. with a 3-ft. diameter coverage. The extinguishing fluid used is a non-conducter of electricity.

Gwish is sold in cartons of one dozen extinguishers.

Deb Chemical Proprietaries, Ltd., Belper, Derbyshire.

Lead Alloy for Chrome Plating Industry

ANTATAC R.1. is a new lead alloy developed by British Lead Mills, Ltd., to counteract a small but increasing number of inexplicable failures in chrome tank linings. Over two hundred different alloys were made up and tested, and of these, the most promising were put into small pilot plants under rigorous conditions. During tests, it was found that Antatac R.1. Alloy was the only lead alloy which was not attacked by chrome plating solutions, even when all the accepted rules for the pro-

tection of linings were deliberately broken, e.g. reversals of current, standing for months in cold solution, boiling away the solution, addition of impurities. Antatac R.1. is now available in the form of sheet for linings, pipe for heating and cooling coils, and anodes, and is in use in Vauxhall Motors' new plating plant at Luton.

British Lead Mills, Ltd., Byron House, 7–9 St. James's Street, London, S.W.1.

Pretreated Steel Sheet for Vitreous Enamelling

A NEW precoated steel sheet for vitreous enamelling is announced by John Summers and Sons, Ltd. Known as Nitec, it is essentially a basic open-hearth enamelling grade sheet-steel, which, after annealing and cleaning, is coated with nickel and a protective coating of zinc.

Costing approximately £8 per ton more than ordinary vitreous enamelling sheet steel, Nitec eliminates several stages in the vitreous enamelling process It need only be " pickled " for a period of three minutes, during which time the zinc coating is removed and the steel degreased. The removal of the zinc coating exposes the nickel flash ready for enamelling, and no further operations are necessary, so that the processing times are appreciably reduced.

It is claimed that Nitec will give a more consistent finish than has been possible hitherto, and will reduce the number of rejects, and that adhesion will be better and more reliable. One very important development concerns the single-coat finish, where the necessity for a ground coat is obviated. Tests already carried out have shown that a " self-mottled " coat can be applied directly to the sheet.

The protective coating will have the advantage of preventing the rusting of the sheets during storage ; cleaning will be easier ; and the sheets and semi-finished parts may be kept in store for some time without deterioration. The zinc film has a number of functions : it protects the prepared nickel film from damage ; acts as a protection during transport and storage ; and offers a kind surface to tools, thus enhancing tool life. Nitec can be welded by all accepted methods, and welding conditions are not much different from those for uncoated mild steel.

John Summers & Sons, Ltd. Shotton, Chester.

Vertical Forced-Air Circulating Furnace

THE Hedin vertical forced-air circulation furnace has undergone many internal modifications to raise its efficiency and simplify its construction. The centrifugal-type fan is of sufficient size to force large quantities of air under high pressure through the densest charge, the work container being so situated that all the airflow is directed through the charge. The speed and volume of the airflow result in only a very small drop in temperature from top to bottom in coming up to temperature, and practically no difference when on control. Overheating the outside of the charge by direct radiation is impossible.

Element replacement is effected by merely lifting out the air guide (a few minutes' work) to reveal the elements. The refractories are designed to enable the elements to be removed with minimum bother, but at the same time the elements cannot become accidentally dislodged from their housing.

Highest grade heating elements of heavy-section nickel-chromium wire formed into helical coils are used, and as the elements are not in the air stream, not only are they protected from injury, but the impurities that come off the charge, such as scale, soot and dirt, etc., cannot affect them.

A well insulated cover-lifting mechanism is simple and compact and enables the largest cover to be lifted and swung to either side with minimum effort.

Hedin Ltd., Commerce Estate, South Woodford, London, E.18.

Nimonic 105

HENRY WIGGIN & CO., LTD., announce the introduction of Nimonic 105, a further heat- and creep-resisting alloy in the Nimonic Series. It is a development of the nickel-chromium-cobalt-molybdenum alloy, Nimonic 100, and shows a useful advance in high-temperature properties. It has a greater resistance to creep at very high operating temperatures than Nimonic 100, the minimum life to rupture at 7 tons/sq. in. and 940° C. (1724° F.) being 50 hours.

Alloy	Stress Tons/Sq. in.	Temperature °C.	Hours to Rupture, (minimum)
Nimonic 95	7	940	15
Nimonic 100	7	940	30
Nimonic 105	7	940	50

An important advantage of Nimonic 105 is its improved resistance to a specific type of high-temperature corrosion sometimes experienced in aero and other gas turbines running at elevated gas temperatures. This corrosion occurs in certain conditions of contamination by solid or molten sulphates associated with the combustion process. The fuels used in this temperature range are high-grade distillates, such as aviation kerosene, which at lower temperatures have little or no corrosive effect. It will be appreciated that this form of high-temperature corrosion is quite different from the fuel-ash corrosion troubles met

at much lower temperatures when certain types of boiler oils are burned in gas turbines.

Nimonic 105 has already been tested by the British aero-engine industry and is now in production. The development of this new alloy represents a further advance in the continuing Nimonic story, which began 18 years ago and has since had such a profound influence on high-temperature engineering. Besides being specified for the turbine moving blades of every British jet and turbo-prop engine, the Nimonic alloys are used for many other gas-turbine components, and for a wide variety of industrial uses requiring outstanding strength and durability at high temperatures.

Henry Wiggin & Co., Ltd., Wiggin Street, Birmingham, 16.

Powder Fluxes for Submerged Arc Welding

UNDER the generic name of Muraflux, Murex Welding Processes, Ltd., are producing a group of new powder fluxes for submerged arc welding. Muraflux A, the first of these new fluxes, has been specially developed to meet the need for a high-quality general-purpose flux for the submerged-arc welding of mild steel. It can be used on either A.C. or D.C. supplies with welding currents up to 900 amp., and on all machines suitable for submerged-arc welding.

The flux may be employed with a mild-steel filler wire containing approximately 2% manganese, or with a normal mild-steel wire. Both types of wire, known as Murawire W1 and Murawire W2, respectively, can be supplied by Murex Welding Processes, Ltd. When used with Murawire, Muraflux A produces good-quality welds of a high radiographic standard, and it can be employed for Class I work. In conjunction with Murawire the flux has been granted approval by Lloyd's Register of Shipping and is accepted by the Ministry of Transport.

Muraflux A is suitable for either the single-pass or multi-pass welding of various joints in mild steel, as well as plug welds and the building up of worn mild steel parts. Good penetration can be obtained, and unfused flux can be recovered and used again.

Murex Welding Processes Ltd., Waltham Cross, Herts.

Liquid Level Transmitter

FOXBORO-YOXALL have recently added to their range of pneumatic transmitters a new instrument known as the Type 13FA. It is a direct mounted liquid level transmitter using the 13A force balance design to measure liquid level in open or closed vessels. It transmits an output air signal to standard 3–15 lb./sq. in. receiver recorders and controllers, which may be locally or remotely mounted. The sensing diaphragm is flange mounted to the side of the tank. This means that there are no process piping problems and the 13FA is thus particularly suited for the measurement of difficult fluids of the viscous and slurry types ; e.g., paper pulp, viscose and lime slurry.

The Type 13FA operates equally well on open or closed vessel applications. In open vessel measurement, only the high pressure side of the pressure measuring diaphragm and the raised serrated face of the cell flange are in contact with the process fluid. The low pressure side of the diaphragm is vented to atmosphere. For closed vessel measurement only an equalising leg is

required. If a " wet leg " is used an adjustable range suppressor attachment is employed to give forward reading measurement and to correct for " cold leg " conditions.

The instrument may be used under the most arduous corrosive conditions, and to this end is supplied in three forms : (*a*) with cadmium plated mild steel body and flange, the diaphragm being stainless steel ; (*b*) stainless steel throughout ; and (*c*) body, flange and diaphragm in stainless steel with flange and diaphragm plastic coated. The twin diaphragm measuring element, liquid filled and with a solid stainless steel core, is completely protected against accidental overrange. It covers the full range of the instrument, i.e., 0–20 to 0–850 in. of water, and range changing is a simple matter of moving a range wheel up or down a range rod fitted with a calibrated range scale.

Foxboro-Yoxall, Ltd., Redhill, Surrey.

Weld-Cleaning Fluid

NORTH HILL PLASTICS, LTD., are now in a position to supply Pelox, a new weld-cleaning fluid for use with stainless steel and certain high chromium and nickel alloys. This fluid has proved extremely successful on the continent, and has been tested in the welding laboratories of important research organisations in this country. The liquid is applied to the discoloured areas and the metals immediately polished with a rotating stainless steel brush. The discolouration is removed instantly, leaving a bright matt finish. Any residue from the dried-out liquid is simply removed with a sponge and cold water. The liquid contains no skin irritants and, apart from polishing goggles, gloves and other types of protective clothing are not required.

The makers claim that 1 kg. (2·2 lb.) will clean over 2,000 ft. of weld at a cost of about one-third of a penny per foot. Additional advantages claimed are freedom from continued corrosive attack, because the liquid is self neutralising, and an indefinite storage life.

North Hill Plastics, Ltd., Manley Court, Stoke Newington High Street, London, N.16.

CURRENT LITERATURE

Book Notices

A PRACTICAL GUIDE TO THE DESIGN OF STEEL CASTINGS

52 pp., numerous sketches. Translated from the French and published by The British Steel Castings Research Association, East Bank Road, Sheffield 2. Also obtainable from The Penton Publishing Co., Ltd., 2, Caxton Street, London, S.W.1, and The Penton Publishing Co., Penton Building, Cleveland, 13, Ohio, U.S.A. 12s. 6d.

THE British Steel Castings Research Association, having acquired from the French Editions Technique des Industries de la Fonderie, the English translation rights of their publication known as the " Guide Practique du Tracé des Pièces en Acier Moulé " has now published this valuable work in English, under the title—" A Practical Guide to the Design of Steel Castings." The book deals with steel castings design from the point of view of soundness, economy in production and good engineering design. It is an up-to-date study of the problems of steel castings design and is well illustrated by one hundred and twenty-one working diagrams. It is strongly recommended by the Association as an invaluable *vade mecum* for all designers, draughtsmen, engineers and others—including practical steelfounders and students—concerned with the specification, design and production of cast steel components for all purposes.

CONSTITUTION OF BINARY ALLOYS

By Max Hansen in co-operation with Kurt Anderko. Second edition, 1308 pages, 684 diagrams. New York, 1958, McGraw-Hill Book Co. Inc. $32.50 (£12 12s. 0d.).

THIS unique reference book, which is a complete revision of " Der Aufbau der Zweistofflegierungen " published by Julius Springer, Berlin, in 1936, provides a source of reliable data concerning the constitution of binary alloy systems (phase diagrams) and the crystal structure of metallic phases. Since the publication of the older German text, an enormous amount of information on the constitution of binary alloy systems and crystal structure has been published, the papers being scattered throughout the world's metallurgical, physical, chemical and crystallographic literature. A large number of systems have been more accurately established, and phase diagrams of many additional systems have been determined, such as those of the lesser known elements. Whereas the original covered 828 systems and featured 456 diagrams, the present work includes 1286 systems and 684 diagrams. The number of references has increased from about 5,500 to about 9,600. The literature up to the spring of 1955 has been covered, but numerous papers up to early 1956 have been considered. As regards the deadline up to which the literature could be covered, it varied from the end of 1955 to the autumn of 1957, depending on the position of the system concerned in the alphabetical order. However, the literature of 1956 and 1957 had in most cases to be considered in the form of supplements or notes added in proof ; wherever necessary, additional diagrams were inserted.

Readers familiar with the older German text will find a number of changes in style in the new volume. The mode of presentation had to be altered in view of the increased amount of information available : otherwise the volume would have been not only cumbersome but prohibitively costly. No longer has it been possible to discuss at length the results of different workers, on the grounds of accuracy, reliability, etc., but the authors have analysed and evaluated all the information as compiled in an extensive search, and incorporated their final conclusions in the diagrams, which are, in the main, composite ones. An important difference concerning the diagrams lies in the fact that in the new edition they are based on atomic percentages rather than weight percentages.

The presentation of data concerning the constitution of binary alloy systems would be incomplete if crystallographic data were not considered systematically. As compared with the older work, not only the symmetry of the intermediate phases in given, but their lattice spacings as well. Furthermore, the literature dealing with the variations of lattice parameters of primary solid solutions has also been considered.

Trade Publications

" ABOUT VITREOSIL " is the title of a publication received from The Thermal Syndicate, Ltd., which is believed to be the only reference book on fused quartz and silica in the world. Vitreosil is, of course, pure silica fused into a homogeneous, uniform product containing more than 99·8% SiO_2. It is available in both translucent and transparent forms, the latter transmitting ultra-violet and infra-red radiations as well as visible light. From the production of comparatively small pieces of laboratory ware, processes have been developed and extended until large chemical and similar plants have become regular items of manufacture. Besides detailing the properties of Vitreosil and outlining its applications in various fields, the new booklet discusses the practical aspects of incorporating Vitreosil in research and production equipment, reference being made to cutting and working, cementing, indelible marking, etc.

LEAFLET No. S91, entitled " Series 361 Recorders and Recording Control Stations," is now available from the Publicity Department, Electroflo Meters Co., Ltd. This recorder and control station has many new features—it occupies only 42 sq. in. of panel space and its 4-in. scale offers a greater scale length than many 12 in. circular chart recorders. Up to four measurements (two recordings and two indications) can be given with the recorder or control station, and 22 different model numbers are available for different applications.

IN view of the continued requests for the publication " Modern Malleable " which is now out of print, Hale and Hale (Tipton), Ltd., have produced a new booklet entitled " Malleable Iron by Hale." At one time the malleable cast iron produced in this country was of the whiteheart type, but developments in melting plant, the introduction of control facilities, and improved raw materials during the last few decades have greatly increased the field for blackheart malleable castings and

pearlitic malleable iron, in the production of which great progress has been made. The new publication discusses the characteristics of blackheart malleable iron and its applications, and describes in some detail the methods used in the manufacture of castings of this type at the Hale works at Dudley Port. This interesting and informative publication is available free to apprentices, engineers, buyers, and others interested in blackheart malleable castings.

THE latest issue of *Wiggin Nickel Alloys* (No. 48) provides an interesting demonstration of the versatility of nickel and its alloys. The applications referred to in the numerous illustrated articles range from distillation equipment for the production of fresh water from sea water, through a series of items of chemical plant to uses where heat resisting properties are required, as in the re-entering of a missile into the earth's atmosphere, or the more prosaic waste gas burner. The central "double-page spread" gives particulars of some fifteen standard products of Henry Wiggin & Co., Ltd.

IN a 26-page catalogue (No. 65) on electrical flow meters, available from Electroflo Meters Co., Ltd., details are given of the variety of differential pressure producers marketed by the Company, including thin orifice plates, carrier ring orifice plates, flow nozzles, nozzle entrance venturi tubes, orifice flange assemblies and differential pressure measurements at fan inlets and in rectangular ducts. The electrical transducer flow meter converts differential pressure signals into electrical signals on a 40 VA. circuit, giving remote readings. The high speed integrator may be fitted with a pick-up for data reduction and computing systems. Reference is also made to the new miniature 4 in./4 in. indicators, integrators and recorders, especially suitable for graphic panels. Useful notes are given upon the installation of differential pressure producers for different measurements, as well as information on open-channel meters employed in weirs and venturi flumes.

THE August issue of *The Bonderizer*, published by the Metal Finishing Division of The Pyrene Co., Ltd., features a description of the production of electrical steel strip, provided with phosphate laminate coating which serves as an insulating layer and assists lubrication in subsequent stamping. Other articles deal with Bonderizing for pre-painted strip and the use of Bonderite-Bonderlube treatment in the production of drawn cup needle bearings.

TWO recent publications from the Lincoln Electric Co., Ltd., deal respectively with the L.R. 150 SA 150 amp. package welding unit, recently introduced for use with the Land Rover, and the Lincolnweld fully automatic submerged arc process, which has been further improved to give easier, faster and more efficient operation. Some of the major features are listed in the leaflet.

BETWEEN the board covers of the new G.E.C. Process Heating Data Book can be found much more than catalogued details of the very wide range of G.E.C. products in the process heating field, although these are, of course, illustrated and briefly described. About half of its 144 pages are devoted to the presentation of data—ranging from the Greek alphabet to the properties of metallic elements, and from the properties of dry air to a list of brazing materials and atmospheres—likely to be useful to the heat treatment engineer.

SPACE will not allow the mention of all the items in Evershed Publication No. 057, which describes briefly the range of instruments made by Evershed and Vignoles. More than a third of the leaflet is devoted to the well-known Megger insulation testers, and other instruments described include roll chart recorders; a gauge for measuring the thickness of non-magnetic coatings on ferrous bases; polarographs; and an optical pyrometer.

WE have received from the English Electric Co., Ltd., five new publications. No. ES/134 describes a 3 h.p. motor controller, which provides stepless uni-directional speed control of a D.C. motor from an A.C. mains supply, and maintains the speed substantially constant, irrespective of load changes. The ½-h.p. Magamp motor control unit described in No. ES/133 provides a robust and inexpensive method of controlling a D.C. motor from an A.C. supply, either for speed or tension control. The remaining publications, DM/239, FG/138 and FG/134, describe, respectively, the B.S.D. ventilated motors; the Red Spot heavy duty fuseboard, now available for industrial purposes; and open type sub-station fusegear.

ANALMATIC auto titration equipment is featured in a new Baird and Tatlock leaflet. A full description of the various items which are combined to produce an instrument whereby a wide variety of titrations—including those involving *p*H or dead-spot end points—can be carried out, and the result printed on a recorder.

THE Tube Investments group has been growing apace in recent years, and for that reason alone the publication of "This is T.I. Today" is to be welcomed. Not only are the names and addresses of the firms in the group, or associated with it, set out, but details are given of the range of products of the various firms in the Steel Tube Engineering, Cycle, Iron and Steel, Aluminium, Electrical, and General Divisions.

MOST of the latest issue of *Murex Review* is taken up with an interesting article in which Mr. R. L. Samuel surveys the factors controlling metallic diffusion from the gas phase, with particular attention to chromising and siliconising. Also included in this issue is the first part of an article by Mr. E. C. Piggot on metallurgical absorptiometry and spectrophotometry.

Books Received

Proceedings of the Congress on Modern Analytical Chemistry in Industry held at the University of St. Andrews, 1957. Organised by the Scottish Section of the Society for Analytical Chemistry. 244 pp. Cambridge, 1958. W. Heffer and Sons, Ltd. 42s. net.

"Electric Furnaces." Edited by C. A. Otto. 244 pp. inc. numerous illustrations and index. London, 1958. George Newnes, Ltd. 35s.

"A Handbook of Lattice Spacings and Structures of Metals and Alloys." By W. B. Pearson. Vol. IV of the International Series of Monographs on Metal Physics and Physical Metallurgy. 1044 pp. inc. numerous illustrations and index. London, New York, Paris, Los Angeles, 1958. Pergamon Press. £12. 12s.

"Effects of Radiation on Materials." Edited by J. J. Harwood, H. H. Hausner, J. G. Morse and W. G. Rauch. 355 pp. inc. extensive bibliography and index. New York and London, 1958. Reinhold Publishing Corporation and Chapman & Hall, Ltd. 84s. net.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
OCTOBER 1958 Vol. LVIII, No. 348

The Determination of Cobalt in Iron and Steel

By R. C. Rooney, A.R.I.C., A.C.T.
The British Cast Iron Research Association

A method is described for the rapid determination of cobalt in iron and steel, in the range 0·001% to 1%. By suitable modifications this range can be extended down to 0·00001%, or up to 3%. After preliminary separation of iron and copper by extraction of their cupferrates into chloroform, residual cupferron is destroyed and the cobalt extracted into benzene as its 2-nitroso-1-naphthol complex. Other metallic complexes are destroyed, excess reagent removed, and the cobalt determined spectrophotometrically.

THE determination of small amounts of cobalt in cast iron has attracted little attention until recently, as there is little or no evidence that it has any structural or metallurgical effects on the material. If, however, cast iron or steel is to be used as a structural material under conditions where it will be subject to a high density neutron flux, then the high excitation cross section of this element gives it great importance from the radiation hazard point of view. The work described below was carried out in response to requests for a method for the determination of cobalt at levels below 0·005%, when the gravimetric,[1] volumetric[2] and direct colorimetric[3] methods become inapplicable.

Experimental

Boyland[4] has described the colorimetric determination of small amounts of cobalt by extraction of the coloured complex with 2-nitroso-1-naphthol. He reported that the complex is formed rapidly between *p*H 4 and *p*H 9, and that by shaking for 10 minutes with a solution of the reagent in carbon tetrachloride, the cobalt is quantitatively extracted. The complexes of other heavy metals such as iron and nickel are decomposed by shaking the extract with concentrated hydrochloric acid, excess reagent removed by shaking with N sodium hydroxide, and the colour of the extract measured in a microcolorimeter using a green filter.

It was considered that Boyland's procedure could be improved upon from a manipulative point of view, as the 10 minutes shaking time necessitated the use of a mechanical shaker; also, the use of a heavy organic solvent made it necessary to remove the aqueous phase and the subsequent washings by suction.

Effect of Standing Time

Previous experience of solvent extraction separations in our laboratory has shown that complex formation proceeds much more rapidly in homogeneous solutions than when the complex-forming reagent is added as a solution in an immiscible solvent.

Accordingly, 10 ml. of a 0·1% solution of 2-nitroso-1-naphthol in methanol was added to a number of solutions containing 100 μg. of cobalt, and buffered to *p*H 5·0–5·5. These solutions were then allowed to stand for 0, 2, 4, 6, 8, 10, 15 and 20 minutes, respectively, before extracting the cobalt complex into 20 ml. of benzene by shaking for 30 seconds. The benzene layer was separated, transferred to a 50 ml. beaker and evaporated to dryness. The organic material was destroyed by fuming with nitric and perchloric acids, and the cobalt in the solution determined polarographically. It was found that complete extraction was achieved with 2 minutes standing; no further effect was noted with increased standing time.

The polarographic determination of the extracted cobalt was used throughout the experimental work, as it provided an absolute method of determination which could be standardised against pure cobalt solutions which had not been processed.

It is apparent that the extraction can be carried out satisfactorily in 2–3 minutes, as opposed to the 10–12 minutes required by Boyland's procedure. Benzene can also be used as extracting solvent, which will allow the use of separating funnels to run off the heavier aqueous phase and washings.

Effect of Washing Benzene Layer

Six solutions containing 100 μg. of copper, cobalt, nickel and ferric iron were buffered to *p*H 5, 10 ml. of the methanolic reagent solution added, and the complexes extracted into benzene as before. The benzene extracts were washed with water, concentrated hydrochloric acid, water, N sodium hydroxide and, finally, water. After each wash, one of the benzene layers was evaporated to dryness, treated with nitric and perchloric acids and the cobalt, iron, nickel and copper determined polarographically. The findings agreed exactly with those of Boyland, i.e. shaking with concentrated hydrochloric acid quantitatively destroys the complexes of the interfering metals, while the cobalt complex is unaffected. Washing with N sodium hydroxide effectively removes excess reagent, again leaving the cobalt unaffected. Completeness of extraction was checked and found to be effective down to 0·2 μg., using a cathode ray polarograph and slightly modified reagents in order to avoid zinc and cobalt blanks.

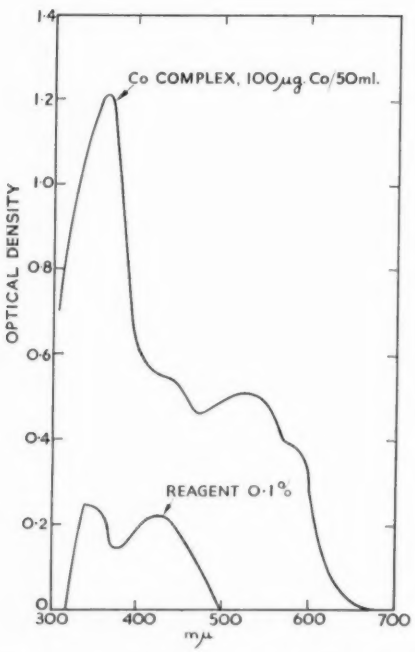


Fig. 1.—Absorption spectrum of cobalt as 2-nitroso-1-naphthol complex.

Spectrophotometry of the Cobalt Complex

The absorption spectra of the cobalt complex and of the free reagent in solution in benzene were plotted, and are shown in Fig. 1. The cobalt complex shows a well defined peak at 365 mμ, and a region of high absorption from 400 to 600 mμ, with a poorly defined maximum at 530 mμ.

The reagent, at much higher concentration, shows a very much smaller peak at 345 mμ, with a region of high absorption from 360 mμ to 480 mμ, with a poorly defined maximum at 430 mμ. Small amounts of reagent left in the benzene solution with the cobalt complex should have a negligible effect on the absorption.

Separation of Cobalt from Iron

Although cobalt and iron can be separated by shaking the mixed complexes with concentrated hydrochloric acid, this method is obviously unwieldy with iron : cobalt ratios of the order of 100,000 : 1, because of the large amount of complexing reagent needed to combine with the iron. A preliminary separation of iron must therefore be made.

Cobalt can be satisfactorily determined spectrophotometrically in the range 0·5 μg. to 300 μg. (see below " Calibration "). If a 0·1 g. sample is used, this corresponds to 0·0005% to 0·3% : by using a sample weight of 0·01 g., this range can be extended to 3%, and, similarly, if a 5 g. sample is used, the bottom limit can be extended to 0·00001%.

It has been shown that 0·1 g. of iron can be easily and rapidly removed by extraction as cupferrate,[5] while larger amounts of iron can be separated from cobalt by ether or ester extraction of ferric chloride.[6]

Preliminary investigations showed that these separations were satisfactory, provided that sufficient acid was present before the cupferron extraction to maintain the *p*H below 1. If insufficient acid were present, the strong buffering action of the cupferron caused the *p*H to rise to about 5–6, and at this level some cobalt cupferrate is formed and extracted, causing low results to be obtained. For the same reason, residual cupferron in the aqueous phase must be destroyed before the cobalt extraction. It was found that 3 ml. of 10N acid was sufficient to keep the *p*H below 1 in the presence of the 25 ml. of 6% cupferron solution used to remove the iron. Using the methods described below, cobalt additions were made to a number of samples of pure iron, and the cobalt recoveries determined. The results are shown in Table I, corrected for blank. The method was then applied to a number of samples. The results are shown in Table II.

Recommended Procedure

Reagents

Very little trouble with reagent blanks has been experienced in the determination of cobalt at the 1 μg. level. It is recommended that reagents should be of AnalaR or equivalent grade whenever possible.

Hydrochloric Acid (sp. gr. 1·16—1·18)

Nitric Acid (sp. gr. 1·42)

Perchloric Acid (sp. gr. 1·54 (60%))

Cupferron (6%) Dissolve 6 g. of cupferron in water and dilute to 100 ml. Filter before use. This solution must be made up fresh as required.

Chloroform (AnalaR)

Sodium Acetate (2M) Dissolve 272 g. of hydrated sodium acetate, $CH_3COONa.3H_2O$ in water and dilute to 1 litre.

Benzene (AnalaR)

Sodium Hydroxide (N) Dissolve 40 g. of sodium hydroxide pellets in water and dilute to 1 litre. Store in a polythene bottle.

2-Nitroso-1-Naphthol (0·1%)–Dissolve 1 g. in methanol and dilute to 1 litre with methanol. Some samples of this reagent may give high blank figures ; in this case the reagent should be recrystallised from hot water.

Standard Cobalt Solution—Dissolve 0·0100 g. of pure cobalt sponge in nitric acid, and make up to 1 litre in a graduated flask

1 ml. ≡ 100 μg. Co

10 ml. of this solution are diluted to 100 ml. to give a solution such that

1 ml. ≡ 10 μg. Co

and 10 ml. of this second solution diluted to 100 ml. again, to give a solution such that

1 ml. ≡ 1 μg. Co

Calibration

Range 1—Measure aliquots of standard cobalt solution equivalent to 0, 20, 50, 100, 150, 200, 250 and 300 μg. of cobalt into a series of 100 ml. conical separating funnels and add 1·0 ml. of hydrochloric acid and 20 ml. of sodium acetate solution to each. Shake, add 10 ml. of 2-nitroso-1-naphthol solution, shake again, and allow to stand for 2 minutes. Add 20 ml. of benzene, shake for 30 seconds and allow to separate. Run off the aqueous phase and wash the benzene layer by shaking for 10–20 seconds each with :—

(1) 10 ml. of water

TABLE I.—DETERMINATION OF COBALT ADDITIONS TO PURE IRON.

Sample Weight (g.)	Cobalt Added (μg.)	Cobalt Found (μg.)		
0·1	1·0	1·1	1·3	1·1
0·1	10	10·2	10·6	10·4
0·1	50	49·9	50·0	50·0
0·1	100	100	101	100
0·1	300	300	300	297
3	1·0	1·4	1·3	1·6*
3	10	10·2	10·4	10·1
3	100	101	105	108

* This figure is so much lower than the blank from the iron that it is of little value.

TABLE II.—DETERMINATION OF COBALT IN IRON AND STEEL SAMPLES.

Sample	Cobalt (%)			
	Certificate Value	Determined Value		
S811*	0·012	0·015 ;	0·015 ;	0·013
S812*	0·25	0·23 ;	0·24 ;	0·24
S813*	0·021	0·021 ;	0·022 ;	0·023
S814*	0·19	0·18 ;	0·19 ;	0·18
S815*	0·06	0·058 ;	0·062 ;	0·061
S816*	0·027	0·026 ;	0·025 ;	0·025
S817*	0·125	0·11 ;	0·13 ;	0·12
Commercially Pure Iron	—	0·0022 ;	0·0024 ;	0·0022
Spectro Pure Iron	—	0·00049 ;	0·00050 ;	0·00049

* Bureau of Analysed Samples Spectographic Standards.

(2) 10 ml. of hydrochloric acid

(3) 10 ml. of water

(4) 10 ml. of N sodium hydroxide

(5) 10 ml. of water.

Reject all the aqueous washings, and dilute the benzene phase to 50 ml. in a graduated flask. Filter through a dry 5·5 cm. No. 541 filter paper into a 1 cm. spectrophotometer cell, and measure the optical density at 530 mμ against benzene. Plot a calibration curve, subtracting the blank reading from all the others in order to make the line pass through the origin.

This graph had a slope such that 0·1 optical density was equivalent to 21·5 μg. of cobalt. The useful range of the graph was thus about 20–300 μg. of cobalt. This range can be extended to 600 μg. by diluting to 100 ml. with benzene instead of 50 ml., or used to cover the range 8–120 μg. more accurately by measuring the original 20 ml. of benzene from a burette and not diluting further, as used in the lower range calibration curve.

Range II—Measure aliquots of standard cobalt solution equivalent to 0, 2, 5, 10, 15, 20, 25 and 30 μg. of cobalt into separating funnels, and process as before. Measure the 20 ml. of benzene from a burette, however, and do not dilute to 50 ml. before measuring. Instead, filter on a dry 5·5 cm. No.541 filter paper into a dry 4 cm. cell, and measure the optical density at 530 mμ against benzene.

Again plot a graph after subtracting the blank value. This graph had a slope such that 0·1 optical density was equivalent to 2·15 μg. cobalt, and covered the range 2–30 μg.

Range III—Process exactly as for Range II but measure the optical density at 365 mμ. This graph had a slope of 0·1 optical density, equivalent to 0·84 μg. of cobalt, and so covered the range 0·5–15 μg. cobalt.

Method for Cast Iron and Steel

A. Cobalt Contents of 0·001–3%: Weigh 1 g. into a 150 ml. squat beaker, and dissolve without heating in 25 ml. of hydrochloric acid and 5 ml. of nitric acid. When dissolved, cool and dilute to 100 ml. in a graduated flask.

Allow the residue of graphite and silica to settle, and pipette a 10 ml. aliquot of the supernatant liquid into a 100 ml. conical separating funnel. (For cobalt contents in the range 0·3–3%, dilute to 500 ml. and pipette out a 5 ml. aliquot : add 5 ml. of water and 3·0 ml. of hydrochloric acid).

To the solution in the funnel, add 25 ml. of 6% cupferron solution, and shake. Add 30 ml. of chloroform, shake for 30 seconds and allow the two phases to separate. Rinse the stopper and neck of the funnel with chloroform from a polythene wash bottle in order to remove any particles of cupferrate adhering to them, and run off the chloroform layer. Check for completeness of precipitation by adding a few drops of cupferron solution to the funnel. A white precipitate of free N-nitroso phenylhydroxylamine should be obtained. If the precipitate is coloured, add a further 5 ml. of cupferron solution.

Add 10 ml. of chloroform, shake for 10–20 seconds and allow the phases to separate. Run off the chloroform layer, and continue to wash with 10 ml. portions of chloroform until the chloroform layer is colourless. Two 10 ml. portions are usually sufficient together with 25 ml. of cupferron solution and the initial 30 ml. extraction. Reject all the chloroform extracts.

Pour the aqueous solution out of the neck of the funnel into a 100 ml. squat beaker, washing with water, in order to avoid contamination of the solution with the ferric cupferrate in the funnel stem. Evaporate the solution to dryness, and add 5 ml. of nitric acid and 3·0 ml. of perchloric acid. Keeping the beaker covered with a well fitting lid, evaporate to fumes of perchloric acid and fume until all organic matter has been destroyed. Cool, and transfer again to a 100 ml. conical separating funnel, washing with water. Adjust the volume to about 25 ml.

Add 25 ml. of 2M sodium acetate solution, and 10 ml. of 0·1% 2-nitroso-1-naphthol solution for every 3 mg. of cobalt plus nickel present. Allow to stand for 2 minutes, add 20·0 ml. of benzene from a burette, and shake for 30 seconds. Allow the two phases to separate.

If much nickel is present, the mixed nickel-cobalt complex does not dissolve readily in benzene, and forms a scum at the interface. If this occurs, add 20 ml. of hydrochloric acid, and shake for 30 seconds, when the mixed complex is destroyed and the cobalt dissolves in the benzene layer. Run off and reject the aqueous phase, and wash the benzene phase with water, acid, water, alkali and water as for the calibration curves. Measure the optical density in a volume of 20, 50 or 100 ml., using the cell size and wavelength appropriate to the amount of cobalt present. The following colours in the 20 ml. benzene layer may be taken as a guide to the amount of cobalt present :

Colourless, or nearly so	less than 5 μg.
Very pale pink	less than 15 μg.
Pink to red	20–100 μg.
Deep red	over 100 μg.

B. Cobalt Contents 0·00001–0·006%: Weigh 5 g. of sample into a 400 ml. squat beaker, and dissolve without heating in 30 ml. of hydrochloric acid and 10 ml. of nitric acid. When dissolved, filter if necessary through a glass wool pad into a 250 ml. conical separating funnel. Wash with concentrated hydrochloric acid from a polythene wash bottle, keeping the volume below 100 ml. Add 150 ml. of iso-butyl acetate, and shake for 30 seconds. Allow the two phases to separate, and run the acid layer into a 150 ml. squat beaker. Evaporate almost to dryness, and add 5 ml. of nitric acid and 5·0 ml. of perchloric acid. Evaporate to fumes of perchloric acid, and fume gently

for 2–3 minutes with the beaker covered. Cool, and transfer to a 150 ml. separating funnel, keeping the volume below 25–30 ml. Add 25 ml. of cupferron solution and extract the iron as in Method A. Transfer to a 100 ml. squat beaker, add 10 ml. of nitric acid, and again evaporate to fumes of perchloric acid, keeping the beaker covered with a well fitting lid. Fume until all the organic matter has been destroyed, and cool. Transfer to a 150 ml. separating funnel, and complete the determination as in Method A.

Acknowledgment

The author wishes to thank the Director and Council of The British Cast Iron Research Association for permission to publish this work.

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3 B. S. 1121 : Part 30 : 1954.
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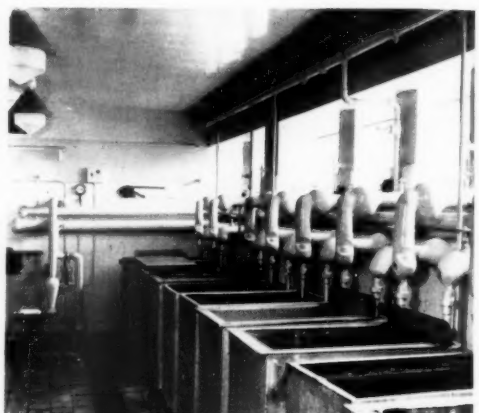
6 Sandell, E. B., "Colorimetric Determination of Traces of Metals," 1950, Interscience Publishers Inc., New York. See especially p. 273.

Technical Service Laboratory for I.C.I. Paints Division

A NEW Technical Service Laboratory building has been erected at Slough to provide the Paints Division of I.C.I. with facilities for giving comprehensive technical service to its customers. This service includes advice on painting specifications and techniques of application, demonstrations to customers, and the training of their painting operatives. Practical trials of new products and new techniques are also carried out in this building.

Closely linked with the Technical Service Department is the Colour Advisory Department, where specialists advise on the use of colour and prepare colour schemes to suit individual customer's requirements. The new building was designed as a main single storey laboratory block with a two storey administration wing, the latter housing the managerial and administration staffs of the Technical Service Department on the ground floor and the Colour Advisory Department above.

In the new laboratory, all the different sections of Technical Service at Slough will be housed under one roof for the first time in the history of the Division. The greatly improved equipment will provide the Paints Division with what is almost certainly the most outstanding paint-service laboratory in this country, and possibly even throughout the world.



Experimental tanks in Pretreatment Section

Technical Service Laboratories

Pretreatment Section.—Materials for the proper preparation of metal before the application of paint constitute an important group of the Paints Division's products. In the Pretreatment Section these materials can be demonstrated on customers' own articles, and panels are treated for use by other sections of the T.S.L. as well as for the Development and Research Laboratories of the Division. There is a well-equipped laboratory for the trial of new chemical-treatment processes and two tank-rooms in which practical trials and demonstrations of all the various processes for the cleaning and de-rusting of metal, and their subsequent phosphating or other chemical treatment, can be carried out.

Industrial Finishing Section.—The Industrial Finishing Section is concerned with the problems relating to the finishing of all manner of manufactured goods other than transport vehicles. These include domestic equipment for kitchens and bathrooms—refrigerators, water-heaters etc.—machinery and agricultural equipment, electrical products, and many others. In the new Technical Service Laboratory this section includes a main application-shop, with separate rooms for flow-coating and electrostatic application, an oven-room with various stoving devices, a separate room for the training of customers' staff and other personnel, and separate rooms for wood finishing.

Transport Section.—This section covers work for motor manufacturers, car-refinishers, industrial transport vehicles and railways. There are two separate rooms, apart from the facilities for trainees which are shared with the industrial section. The larger room contains facilities for finishing a number of motor-cars at one time, if necessary, although much work is carried out on sections of cars such as doors and wings. There is a totally-enclosed unit comprising a spray-booth and oven in which a large motor-car body can be sprayed under completely dust-free conditions and then passed along rails directly into the oven in which the finish is baked hard. The other transport room is intended for the trial of air-drying synthetic paints used on buses and coaches.

Decorative and Marine Section.—This section provides technical service and practical evaluation in connection with all paints used for the protection and decoration of every kind of building or structure, as well as for ships and boats. Probably the most important function of the section is the practical testing and assessment of building and marine paints from the viewpoint of the user.

An Arc Furnace for Zone Refining Metals

By G. A. Geach and F. O. Jones

A.E.I. Research Laboratory, Aldermaston

The use of a furnace in which the metal to be melted is held in a water cooled copper hearth while heated by an arc from an inert electrode overcomes the difficulty of contamination from the containing vessel in zone refining the more refractory or reactive metals.

DIFFICULTIES are encountered when attempting to treat the more refractory or reactive metals by zone refining, contamination from the containing vessel being one trouble. One way of avoiding this difficulty is to use an arc furnace in which the metal to be melted is held by a water cooled copper hearth while heated by an arc from an inert electrode. This note describes a furnace of this type which has been developed in this laboratory. Since the furnace was built, Hulm[1] has mentioned the use of arc heating for zone melting rhenium, but no details of his apparatus are published. We have learnt that a furnace very similar in type to that described here has been built in a laboratory of the Commissariat à l'Energie Atomique.[2]

The furnace is illustrated in Fig. 1. A water-cooled copper hearth, which can be moved steadily under a fixed tungsten electrode in a controlled atmosphere, holds the specimen in a U-shaped groove. An arc is struck between the specimen and electrode, melting a narrow zone of the specimen, usually about $\frac{1}{2}$ in. long.

A separate depression at one end of the hearth holds a pellet of titanium or other suitable metal, which is heated at the outset of an experiment to getter the argon atmosphere in the furnace. Cooling water is introduced through a fixed tube along the axis of the hearth in such a way that the inflowing stream always enters directly underneath the arc. The water passes out of the hearth along a tube concentric with the inlet tube but fixed to the hearth. This outlet tube is attached to a pulling mechanism which moves the hearth under the electrode

The water cooled tungsten electrode is mounted so that it may slide vertically and in a direction perpendicular to the axis of the hearth. To make this movement possible, the electrode is connected to the furnace vessel through phosphor bronze bellows. The electrode can be fixed at any chosen distance from the specimen.

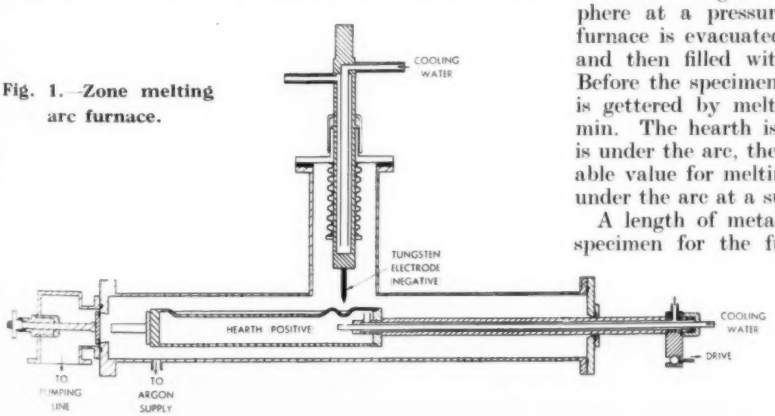


Fig. 1.—Zone melting arc furnace.

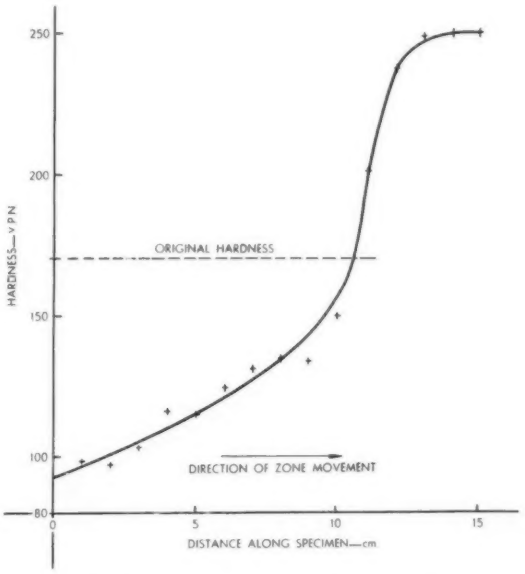


Fig. 2.—Hardness of vanadium after thirty passes of 0·5 in. zone at 0·080 in./min.

The furnace is evacuated by an O2B M-V. diffusion pump, backed by an M-V D.R.1. rotary pump, or filled with gettered argon.

Zone melting is carried out in a purified argon atmosphere at a pressure of about $\frac{1}{3}$–1 atmosphere. The furnace is evacuated, flushed with argon, re-evacuated and then filled with argon to the working pressure. Before the specimen is melted, the furnace atmosphere is gettered by melting a titanium bead for about 10 min. The hearth is then moved so that the specimen is under the arc, the heating current adjusted to a suitable value for melting a zone and the specimen moved under the arc at a suitable rate.

A length of metal rod is the most suitable form of specimen for the furnace, but it is quite simple to use pressed powder pellets or lump material, which can be melted to the shape of the hearth in the furnace before commencing zone melting.

1 Hulm, J. L., *Phys. Rev.*, 1954, **94**, 1390.
2 Cabane, G., *J. Nuclear Energy*, 1958, **6**, 269-274

In some cases it has been found that the arc tends to "wander" during a pass. This is undesirable, but can be controlled by keeping the electrode-specimen spacing as low as possible. Alternatively, the wandering can be suppressed by a magnetic field; use of an Alnico permanent magnet has been found suitable, although a built-in solenoid would obviously be more desirable.

Several metals (for example molybdenum, iron, vanadium) have been satisfactorily zone melted in this furnace. The variation in hardness, which is a measure of the purity, along a zone melted bar of vanadium is shown in Fig. 2.

Acknowledgment

The authors wish to thank Dr. T. E. Allibone, F.R.S., Director of the Laboratory, for permission to publish this note.

British Polarographic Research Institute

IN view of the growing demand for the services of specialists in the various fields of science and technology to-day, the present acute shortage of trained and experienced scientists is creating a problem of the greatest magnitude, especially in the realm of chemistry, where, in spite of the development of analytical instruments as time-saving aids in laboratories and process plants, there is still an unsatisfied demand for chemists skilled in the operation of these instruments and in the interpretation of data. This is no less true in the specialised science of polarography, since the present shortage of skilled and semi-skilled polarographers is depriving this country of the benefits obtainable by the use of this unique analytical tool.

In other parts of the world this shortage has been met by the formation of groups of experts freely offering their services to science and industry in order to ensure that the material rewards arising from the use of this technique are available to all. Notable examples of this type of specialist organisation are the Central Polarographic Institute of Czechoslovakia, established in Prague by Professor J. Heyrovsky, and the Polarographic Centre established at Padova in Italy by Professor G. Semerano. These Institutes are noted for their pioneering work in developing the theory and practice of polarography, and in applying this knowledge to the problems of medicine and science.

Unfortunately, the universal adoption of the polarographic technique in Great Britain has been seriously handicapped by a general lack of appreciation of its potentialities, and by the absence of any central organisation of specialists able to provide practical training in all aspects of polarography, and capable of developing polarographic methods to suit the varied needs of industry. A decision has therefore been made to form a British Polarographic Research Institute for this purpose. Mr. G. F. Reynolds, Principal Scientific Officer, Chemical Inspectorate, Ministry of Supply, has accepted the Chairmanship of the Technical Advisory Panel, whose members are specialists in various branches of polarography, all of whom have offered their services in an honorary capacity.

Training Courses

Full-time and part-time courses in polarography will be available at the Institute during the session commencing November 1958, the object being to provide basic training in the theory and practice of polarography. The courses will include lectures, directed reading and practical exercises in D.C. polarography, A.C. polarography, cathode ray polarography and square wave polarography, and instrumental training will be given on manual polarographs, D.C. pen-recording polarographs, cathode ray polarographs, phase-selective polarographs and square wave polarographs.

Lectures and practical training will be so arranged that attendance on only one or two consecutive days per week is necessary, but some private study is desirable. Each course will normally extend over a training period of thirty hours, and a standard of competence in the practice of polarography will normally be achieved in this period. Training may be completed in a period of three weeks, but to obtain maximum value, a period of at least five weeks is desirable. This allows adequate time for private study. Overnight accommodation is available at the Institute, which is situated within three minutes walk of Woking Station, which can be reached from Waterloo in less than half an hour.

The courses will run continuously from November, and further details of these and other aspects of the Institute's activities may be obtained from the Secretary, Mr. W. J. Parker, 55, Oriental Road, Woking, Surrey.

Pure Reagents for Trace Analysis

AN increasing need for chemical reagents in quantity having extremely low limits of impurities, e.g. for use in polarography or for trace analysis, has led to the formation of an Exploratory Committee to investigate the possibilities of preparing specifications for such reagents. The Committee is anxious to obtain as much information as possible about the limits to which impurities can be tolerated and the estimated annual consumption of reagents. All those interested in the subject are invited to write to the Secretary of the Committee: Mr. W. J. Parker, 55, Oriental Road, Woking, Surrey, from whom a special questionnaire form can be obtained.

The Exploratory Committee consists of representatives of the following organisations: The Animal Diseases Research Institute, The Association of Public Analysts, The British Polarographic Research Institute, The D.S.I.R. Interservices Panel for Physico-Chemical Methods of Analysis, The Medical Research Council (Department for Research in Industrial Medicine), The Photoelectric Spectrometry Group, The Polarographic Society.

Factory Extension

THE Peterhead factory of Cleveland Twist Drill (G.B.), Ltd., manufacturers of engineer's cutting tools, is to be extended by an area of some 35,000 sq. ft., which will include both manufacturing and office space, and bring the total floor area at Peterhead to over 100,000 sq. ft. It is hoped the new facilities will be in use within a year.